

Microstructural Characterisation of Partially Melted Alloys by In-situ X-Ray Microtomography

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Outline

- Introduction
- X-Ray Microtomography
- Investigation during partial melting
- Investigation during solidification
- Conclusion

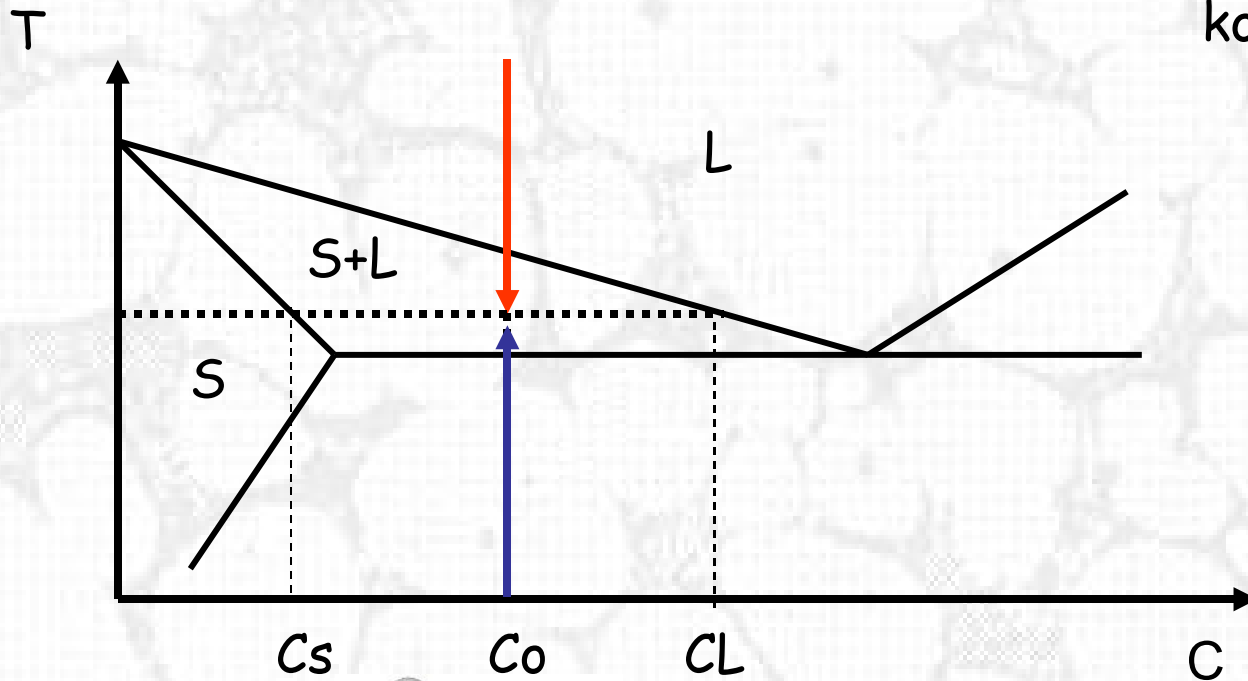
Introduction

Partial melts in alloys

➡ **During solidification**

➡ **During partial melting**

Partition ratio
 $k_0 = C_s / C_L$



Main parameter = solid volume fraction f_s

☞ **Equilibrium: lever rule**

$$f_s = (C_L - C_0) / (C_L - C_s)$$

☞ **No solid diffusion: Scheil equation**

$$C_s = k_0 \cdot C_0 (1 - f_s)^{(k_0 - 1)}$$

Other parameters for the solid

Size

Morphology

Solid-liquid interface area

Connectivity of the solid particles

...

Determination of parameters usually carried out

- after **quenching** of the specimens to room temperature
- on **polished sections (in 2D)**

Questions :

1. Is the solidified microstructure representative of the microstructure in the S/L region?
2. Are observations on 2D sections representative of the 3D microstructure?

Question 1 : Is the solidified microstructure representative of the microstructure in the S/L region?

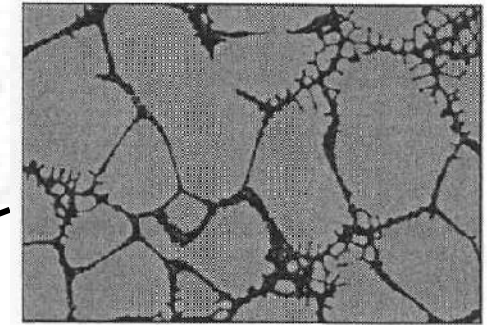
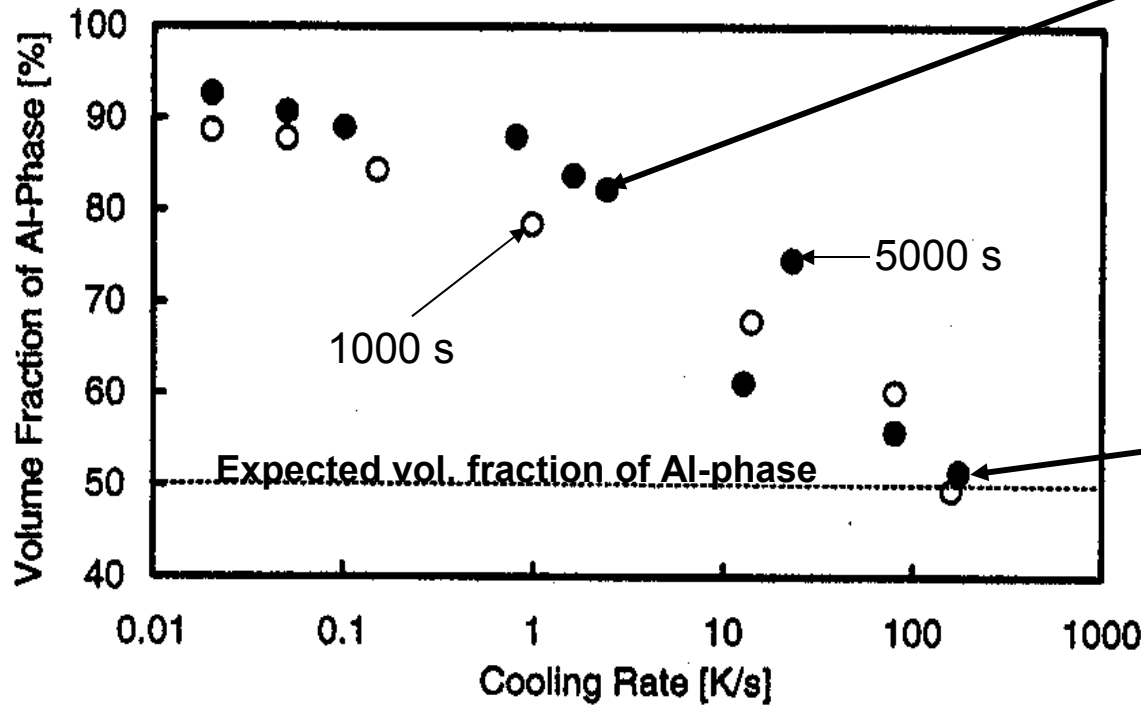
- alloy is just at the eutectic temperature
→ no problem : (liquid → eutectic mixture)
- alloy above the eutectic temperature
→ problem : deposition of solid on the existing solid
and then eutectic transformation
→ solid fraction surestimated

Sometimes etching allows to distinguish the two primary solids (preexisting and that formed during quenching)

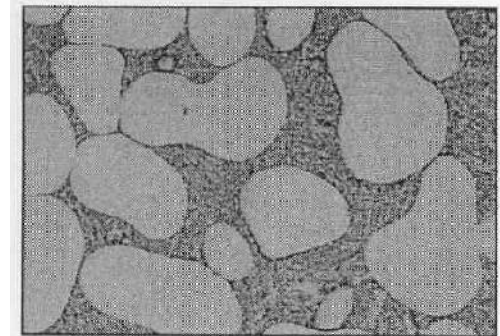
O. Pompe & M. Rettenmayr

J. Crystal Growth, 1998, 192, 300-306

Al-6.8wt%Cu held at 628°C
and cooled at various rates



200 μm



Cooling rate $\geq 200^\circ\text{C/s}$

Question 2 : Are observations on 2D sections representative of the 3D microstructure?

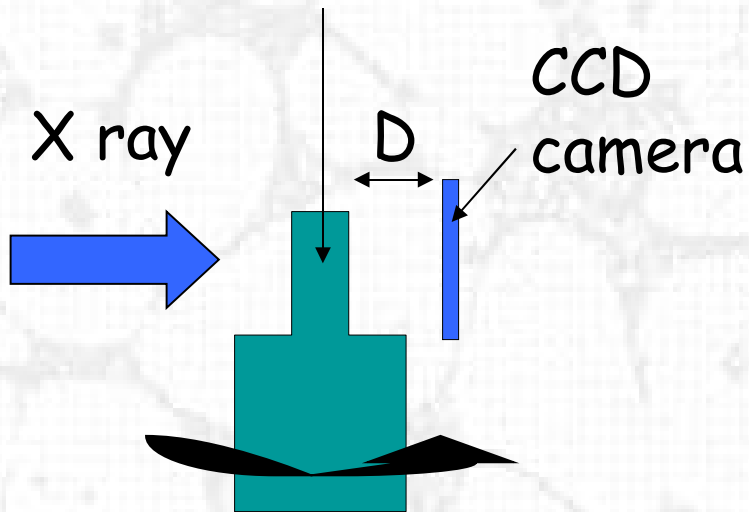
- Yes for some parameters (solid volume fraction, size)
- No for other parameters (connectivity)

Solutions :

- Serial sectioning and 3D reconstruction
destructive
low resolution
but no specific equipment
- X-Ray tomography and 3D reconstruction (computed tomography)
non destructive
high resolution (synchrotron)
but specific equipment

X-Ray Microtomography

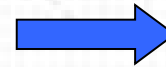
Sample : $\Phi = 2 \text{ mm}$
 $h = 3 \text{ mm}$



~ 900 projections
over 180°

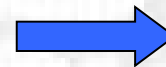
Room temperature

Absorption mode : $D = 3 \text{ mm}$



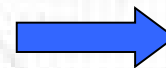
Al-Cu alloys

Phase contrast mode : $D = 500 \text{ mm}$



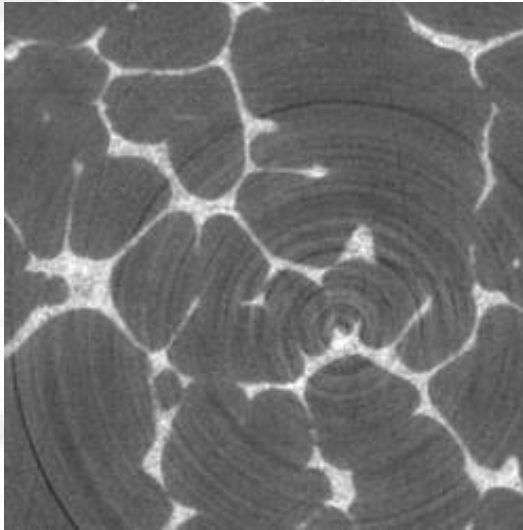
Al-Si alloys

Holotomography mode : several D



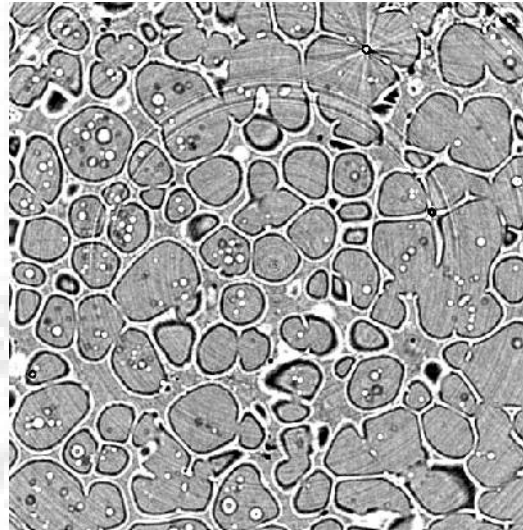
Al-Si alloys

~~150~~ μm



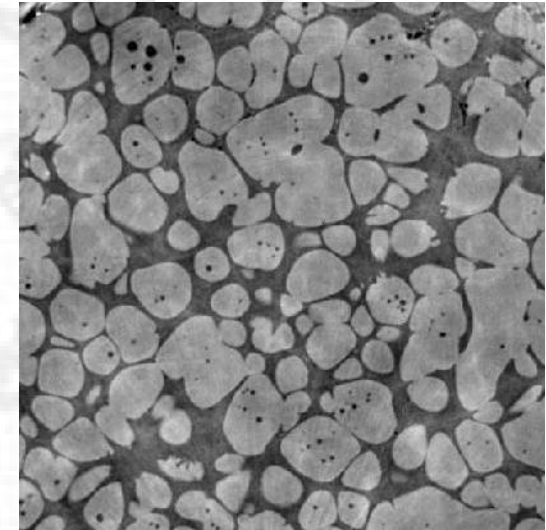
Al-Cu
Absorption

~~100~~ μm



Al-Si
Phase contrast

~~100~~ μm

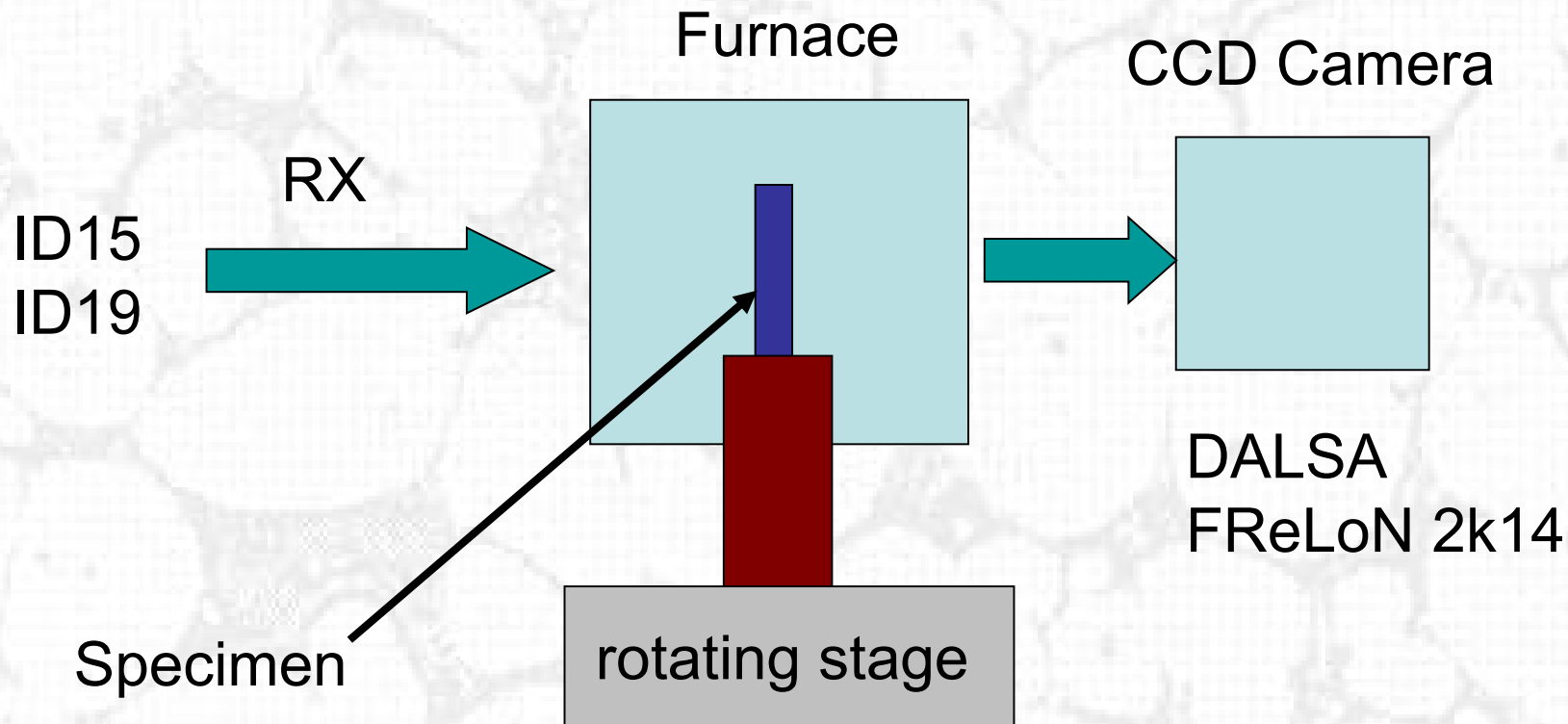


Al-Si
Holotomography

Maximum resolution : $\sim 1 \mu\text{m}$

High temperature

ESRF (European Synchrotron Radiation Facility, Grenoble)



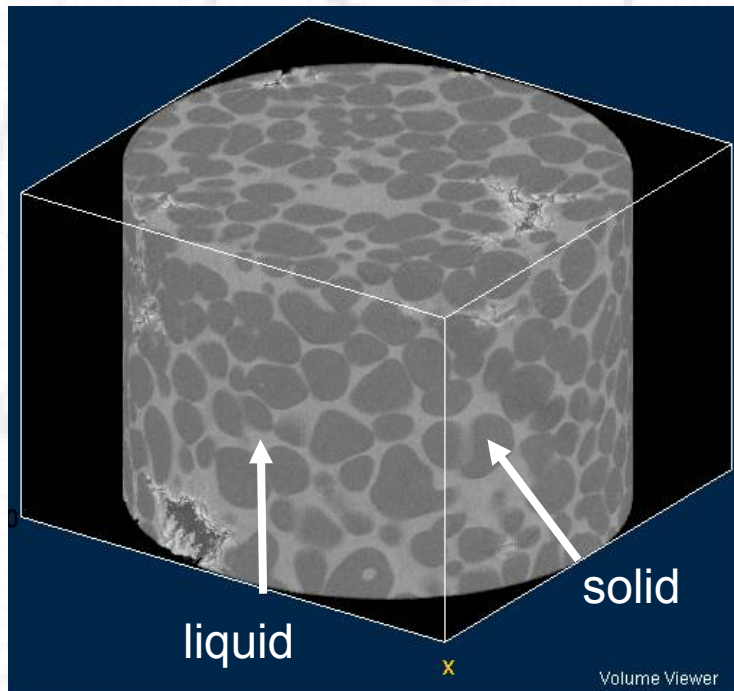
Total acquisition time ~10 to 20s

Limitations

- ☞ Sufficient contrast between solid and liquid phases
- ☞ Small specimens (diameter \sim resolution \times number of pixels)
- ☞ Solid fraction high enough to avoid solid movement during acquisition
 - during solidification $f_s > f_s$ (coherency)
 - during holding $f_s > f_s$ (packing)
- ☞ For solidification, cooling rate slow enough for solid fraction to remain nearly constant during acquisition

Investigation during partial melting

Alloy investigated: Al-15.8%Cu grain refined and rapidly solidified

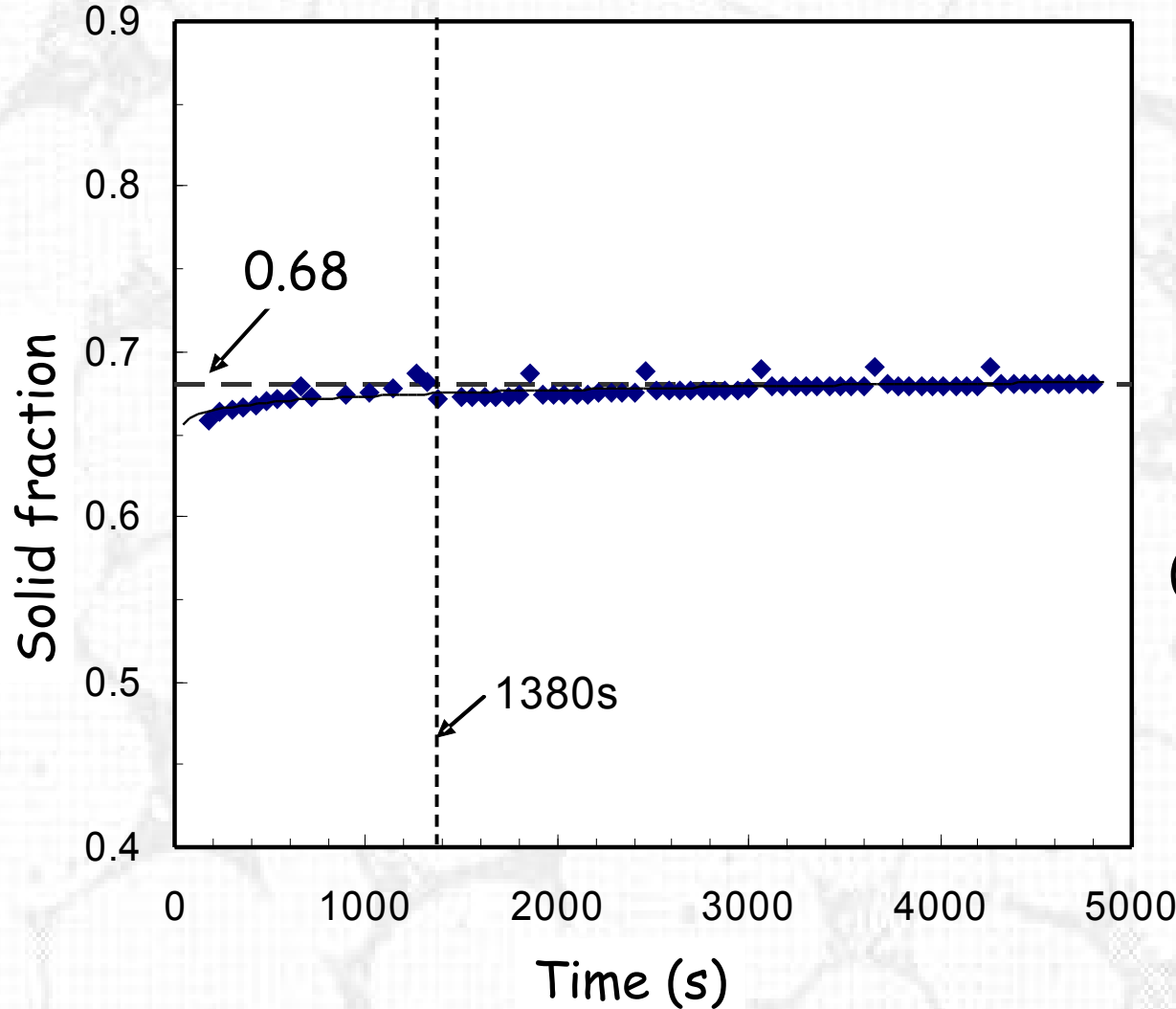


1.5 mm

ID 15
DALSA Camera
Bining
512x512 pixels
400 projections
Resolution: 2.8 μ m
Total scan time: 20s



1.5 mm



f_s increases at the beginning due to diffusion in the solid (return to equilibrium)

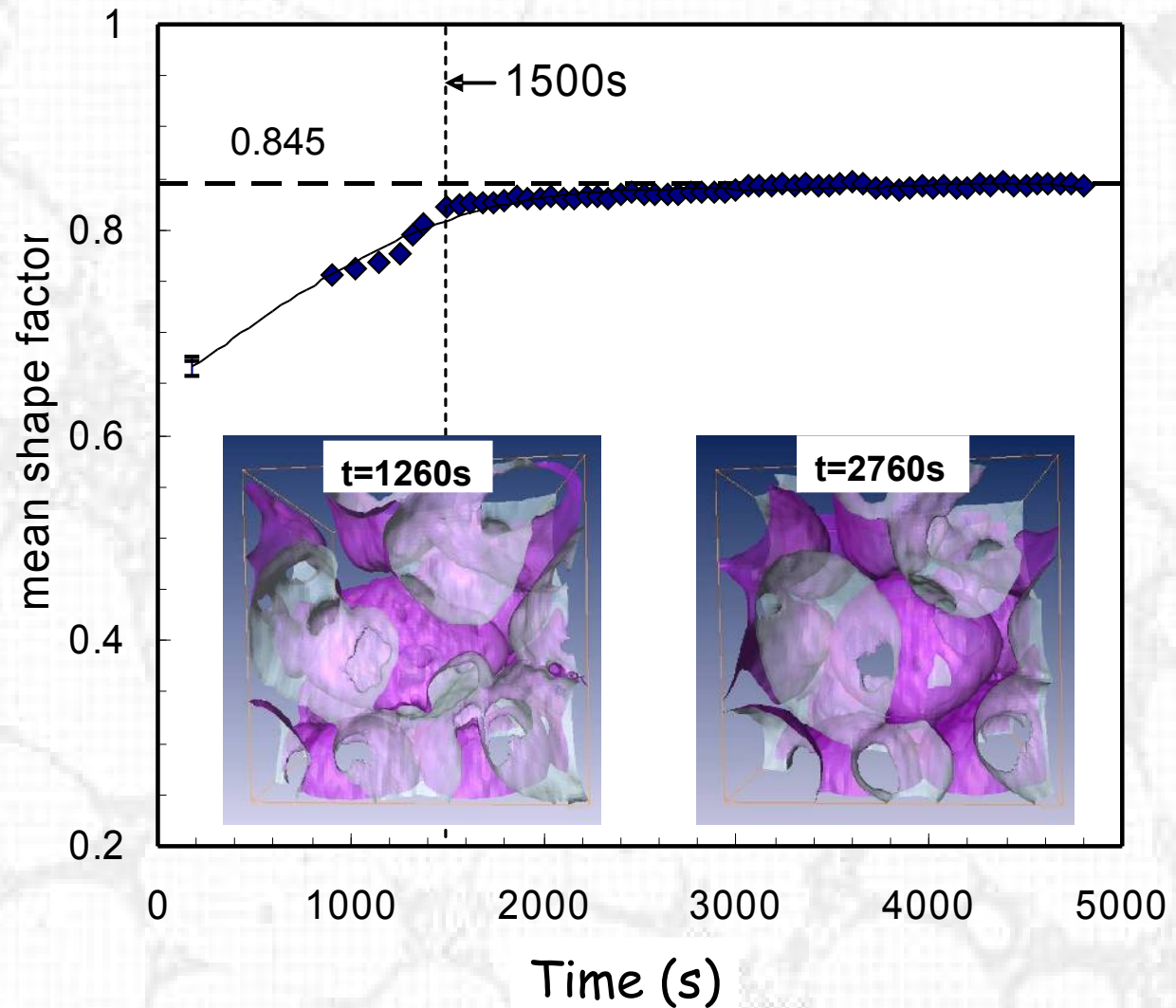
$$s.f. = 6V(\pi/S^3)^{1/2}$$

For a sphere:

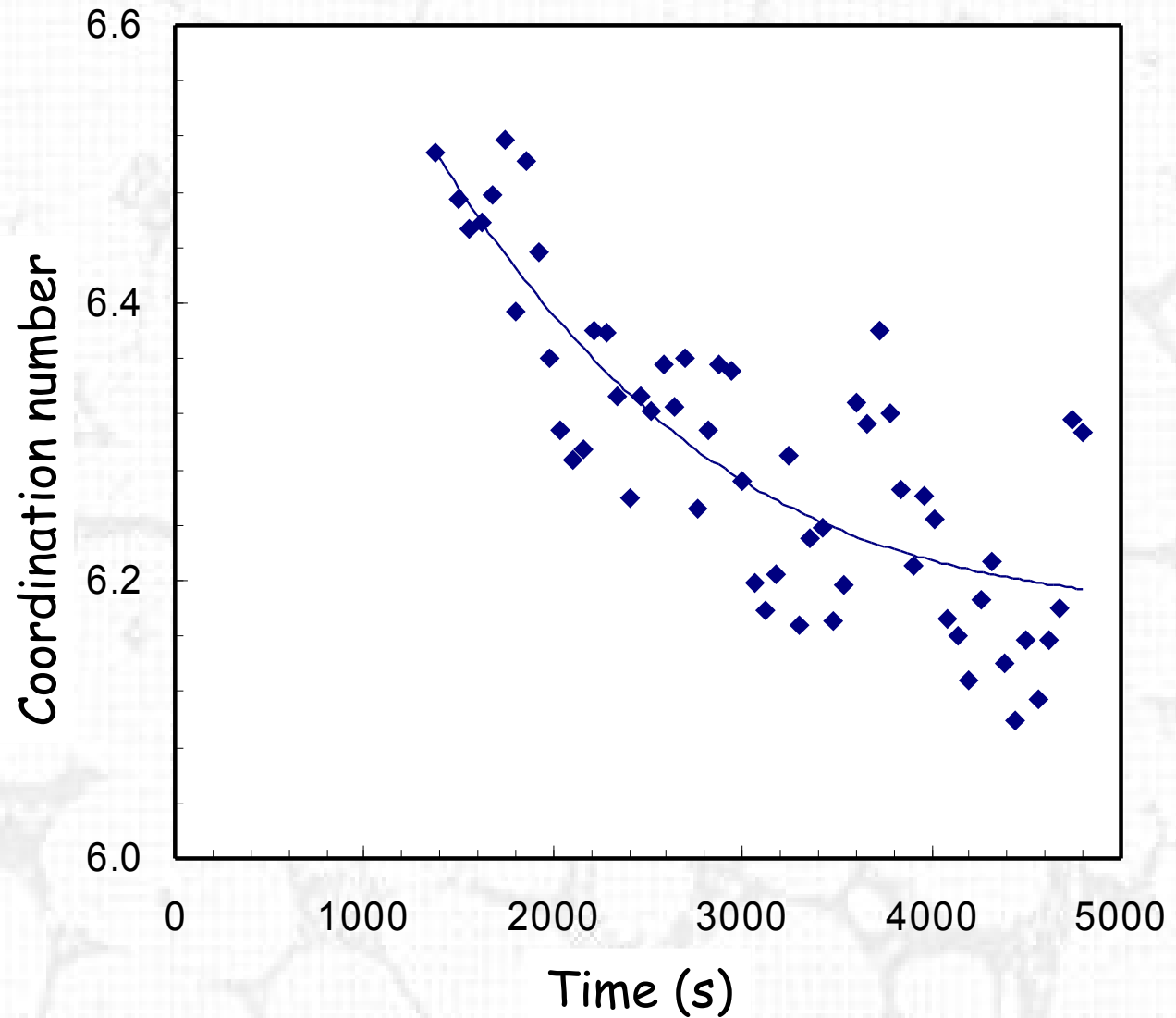
$$V = 4/3 \pi R^3$$

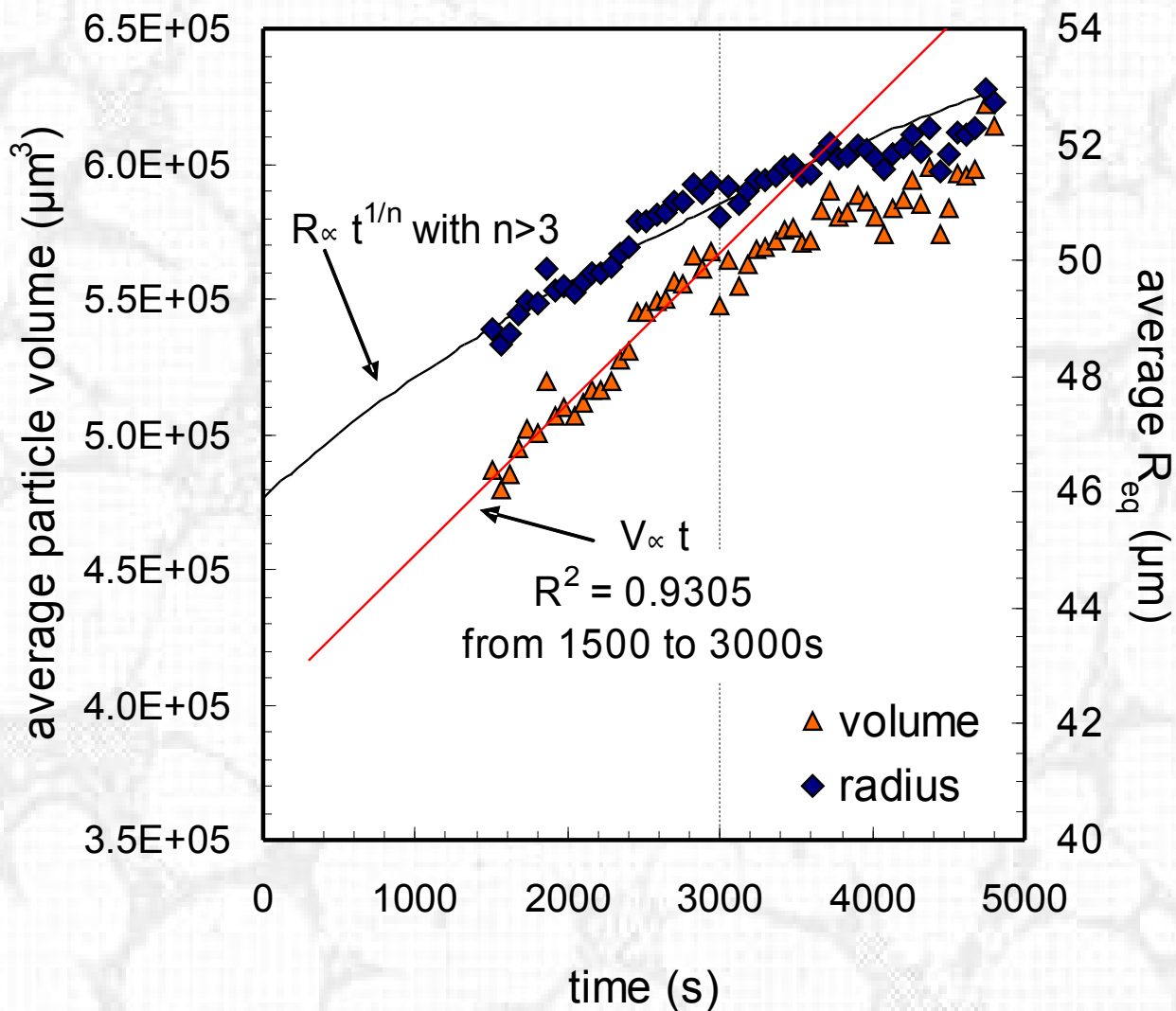
$$S = 4 \pi R^2$$

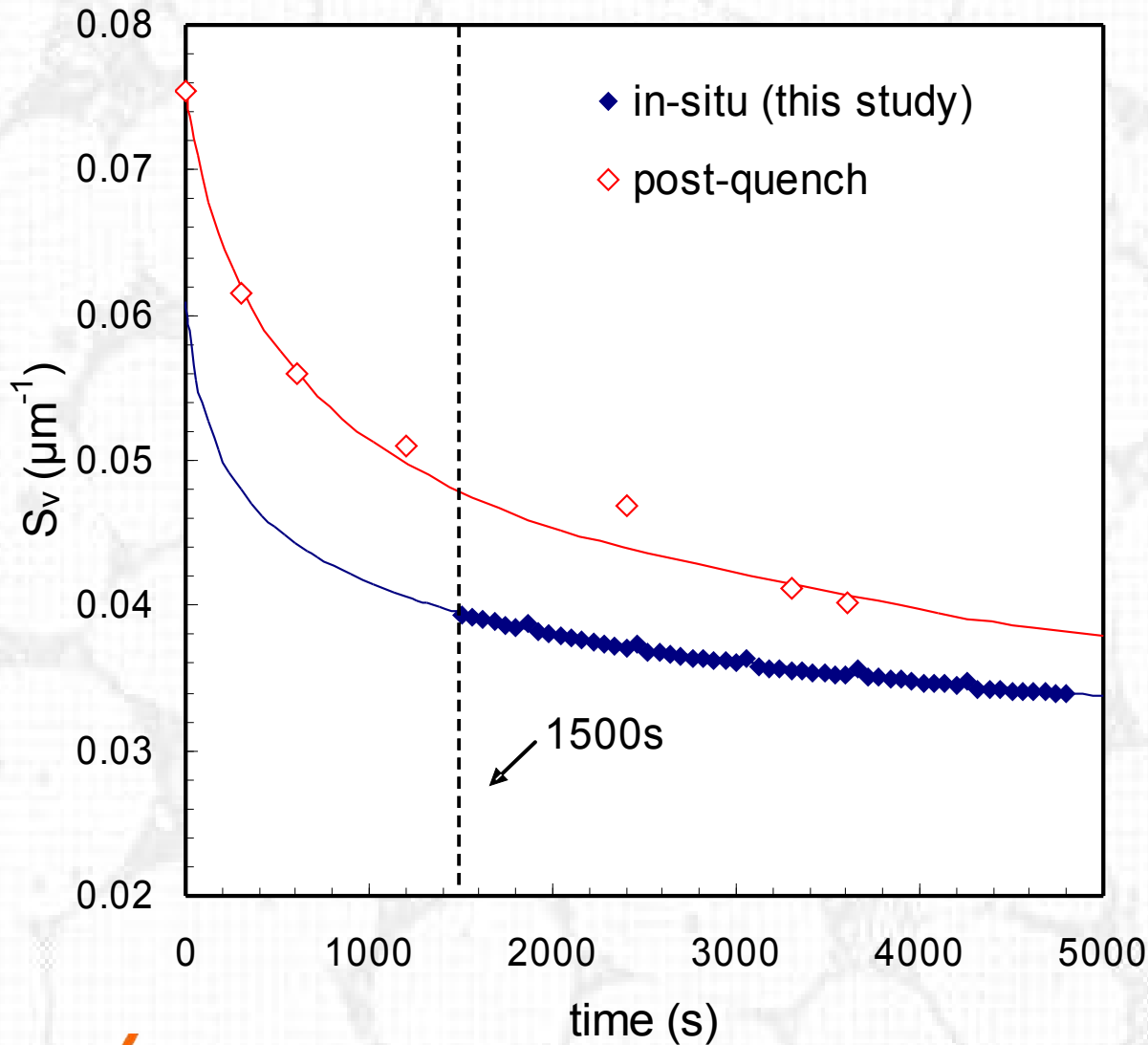
$$\Rightarrow s.f. = 1$$



Coordination number
=
Average number
of neighbours
of a solid
particle



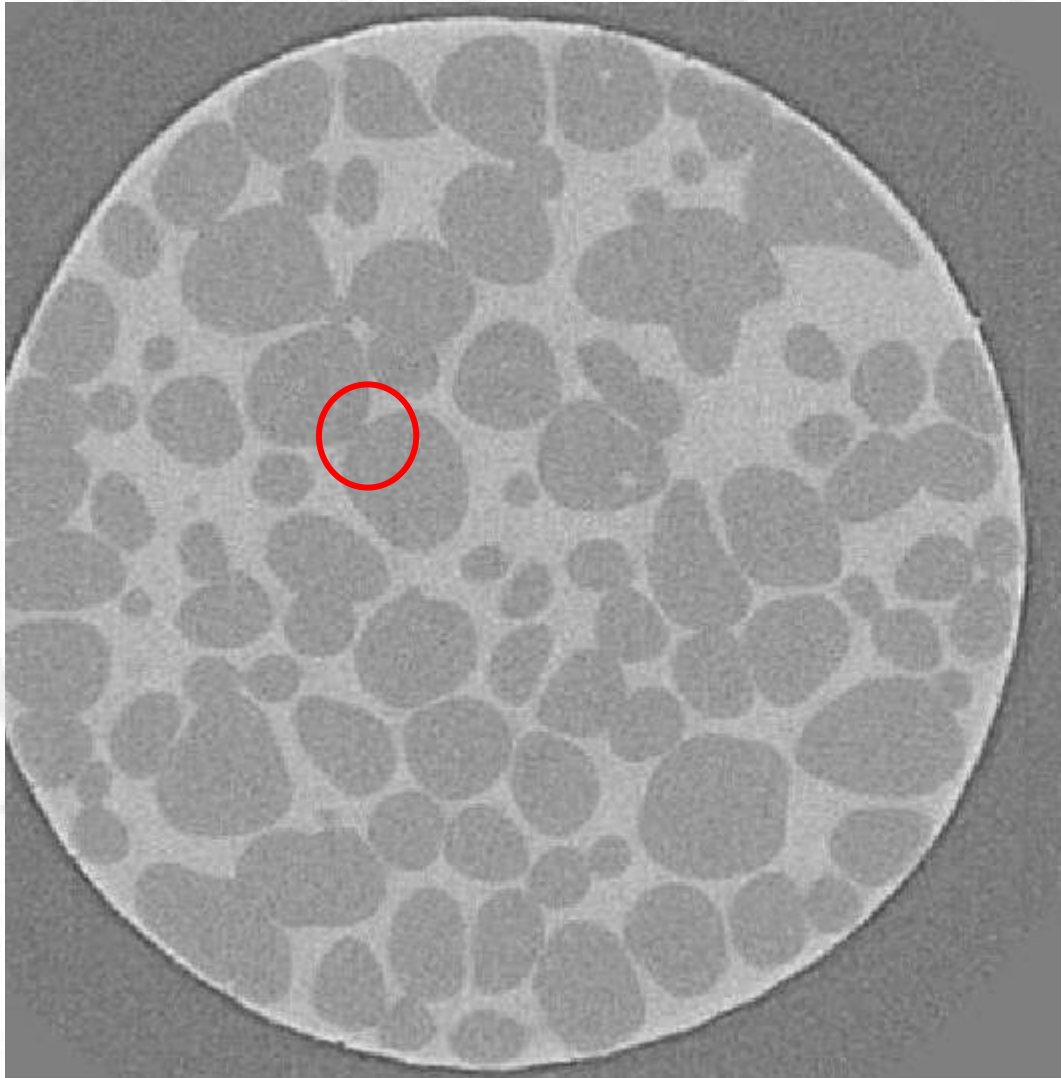




S_v = solid-liquid interface area

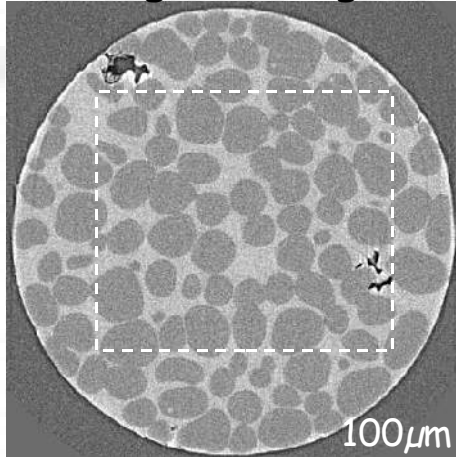
$$S_v = (S_{v0}^{-1/n} + K.t)^{-n}$$

$$n = 1/7.5 \neq 1/3$$

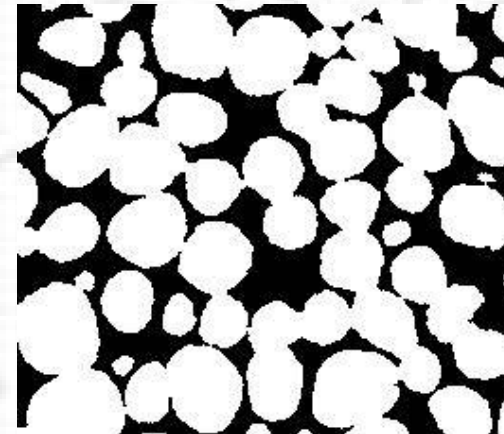


- ☞ Tracking of necks between solid particles
- ☞ 3D quantitative analysis
- ☞ Main mechanisms
- ☞ Comparison with models

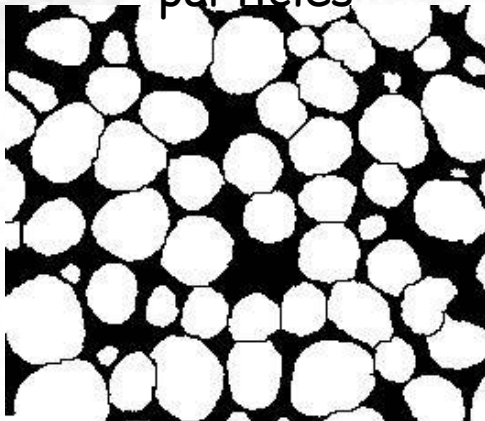
Grey-scale
original image



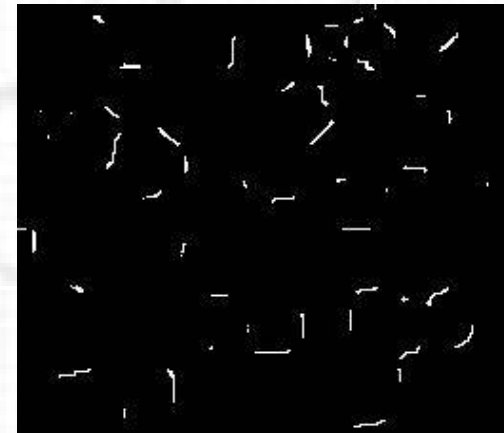
Solid in white
Liquid in black

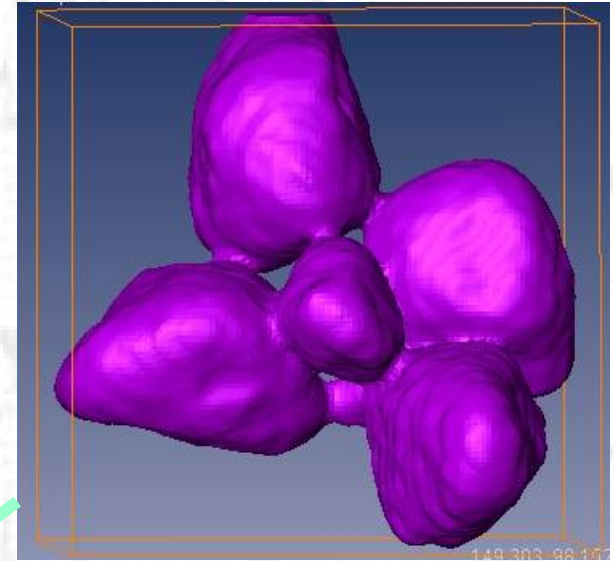
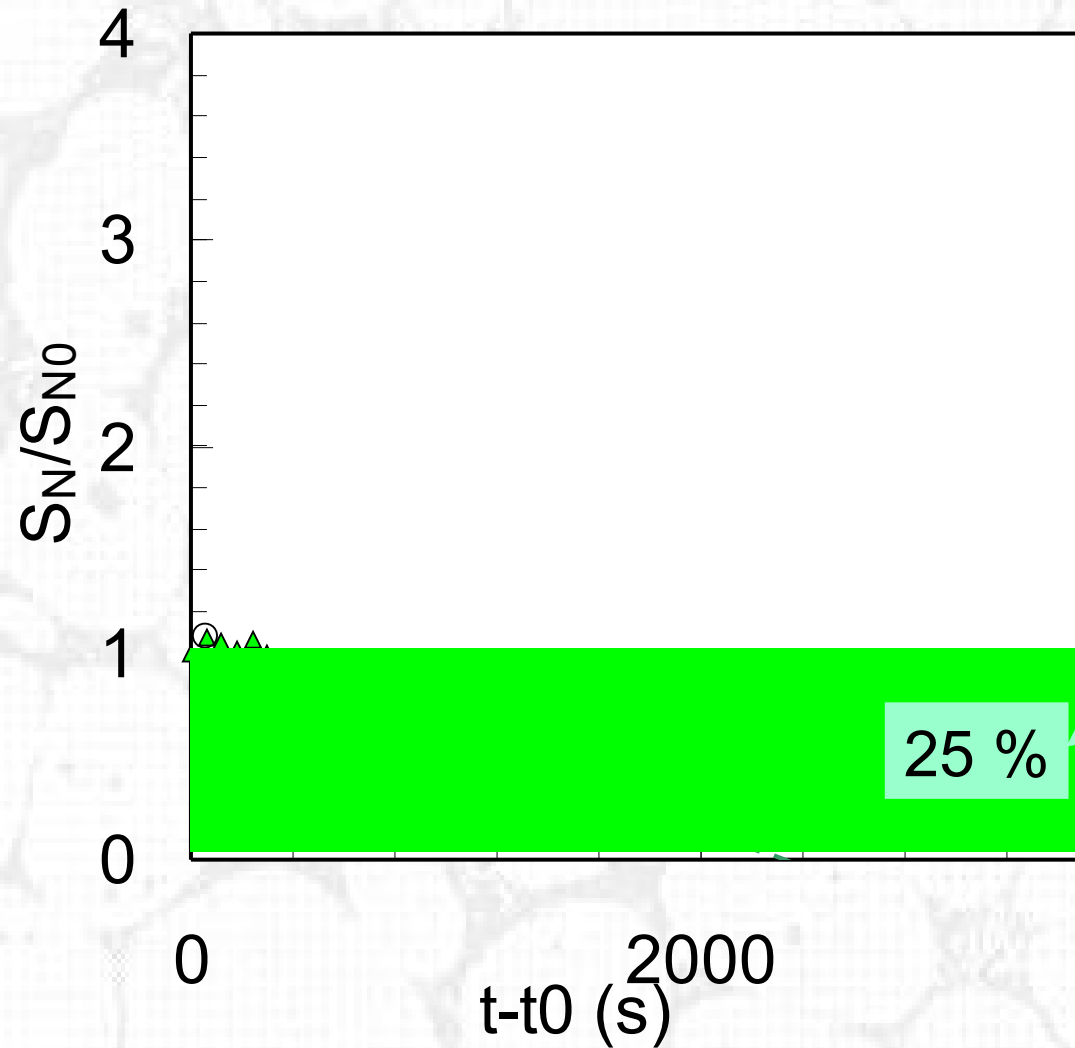


Segmented solid
particles

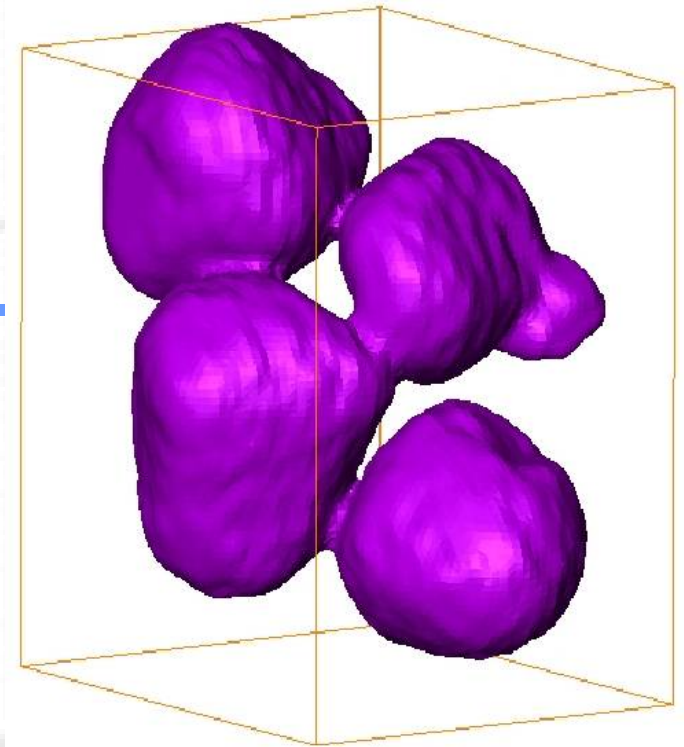
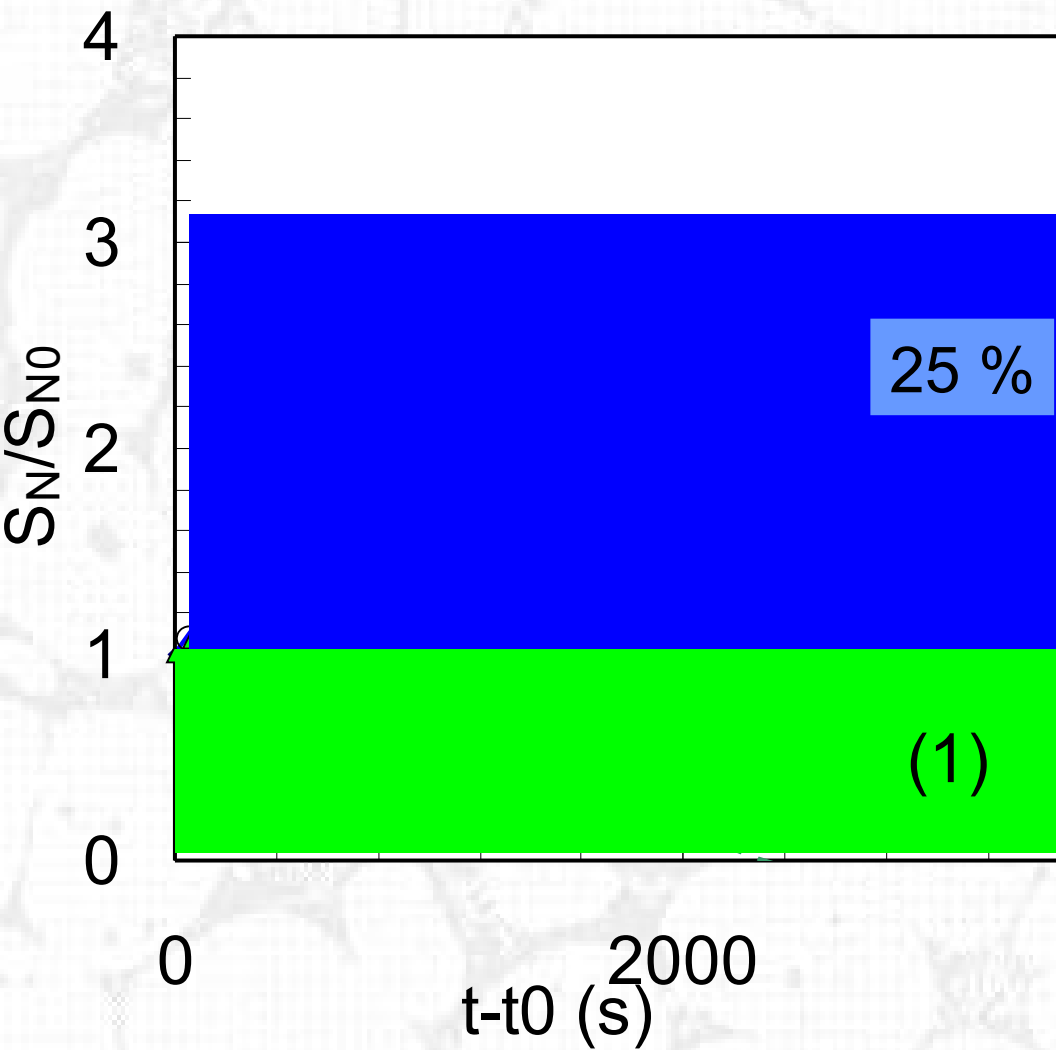


Isolated necks

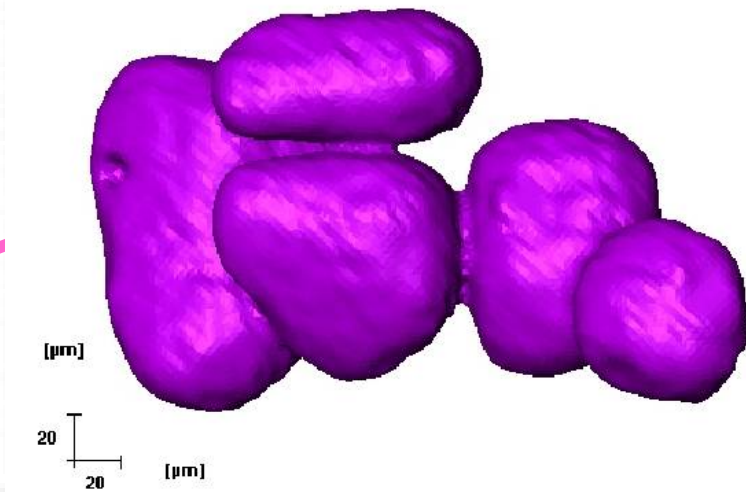
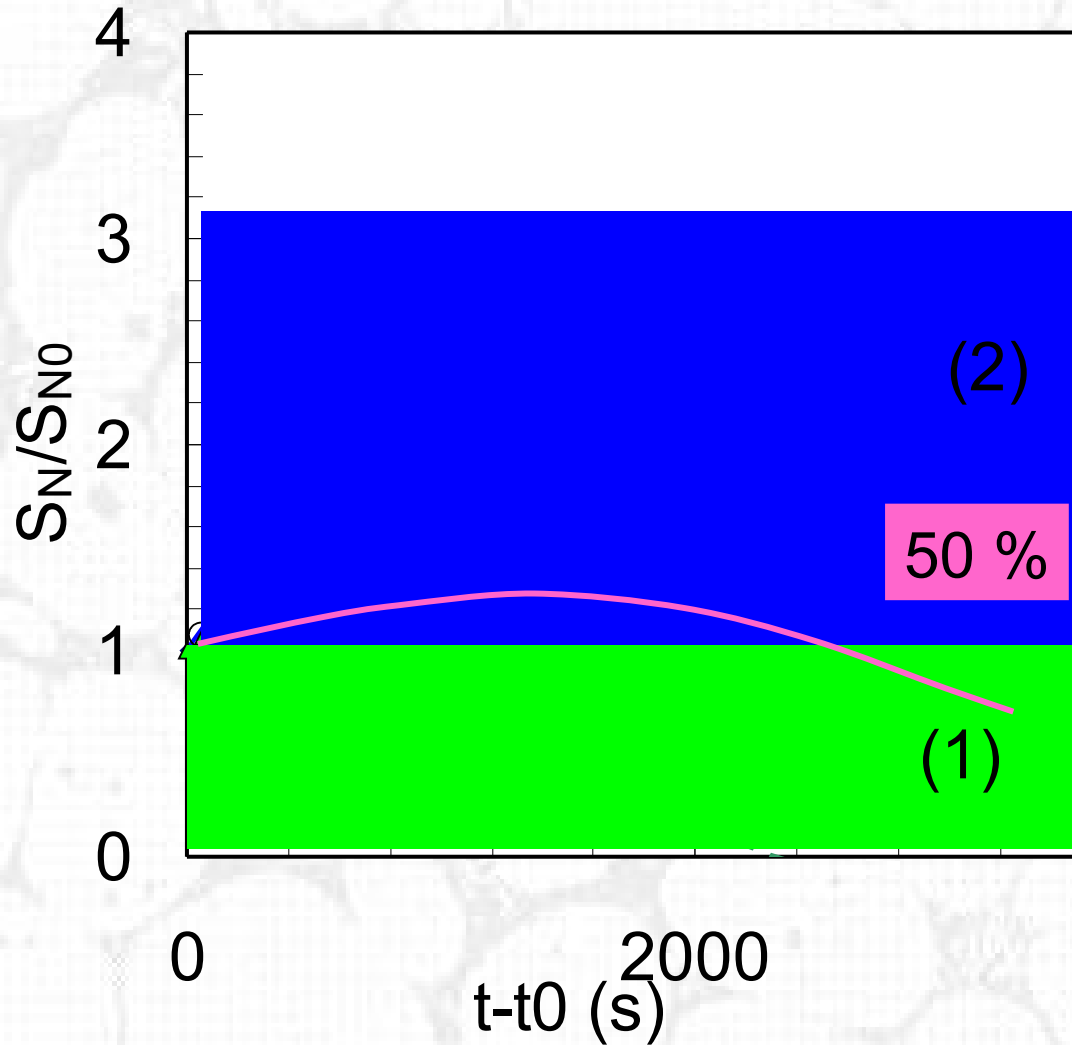




Ostwald ripening
Mechanism



Similar to coalescence
Courtney model
(2 particles)

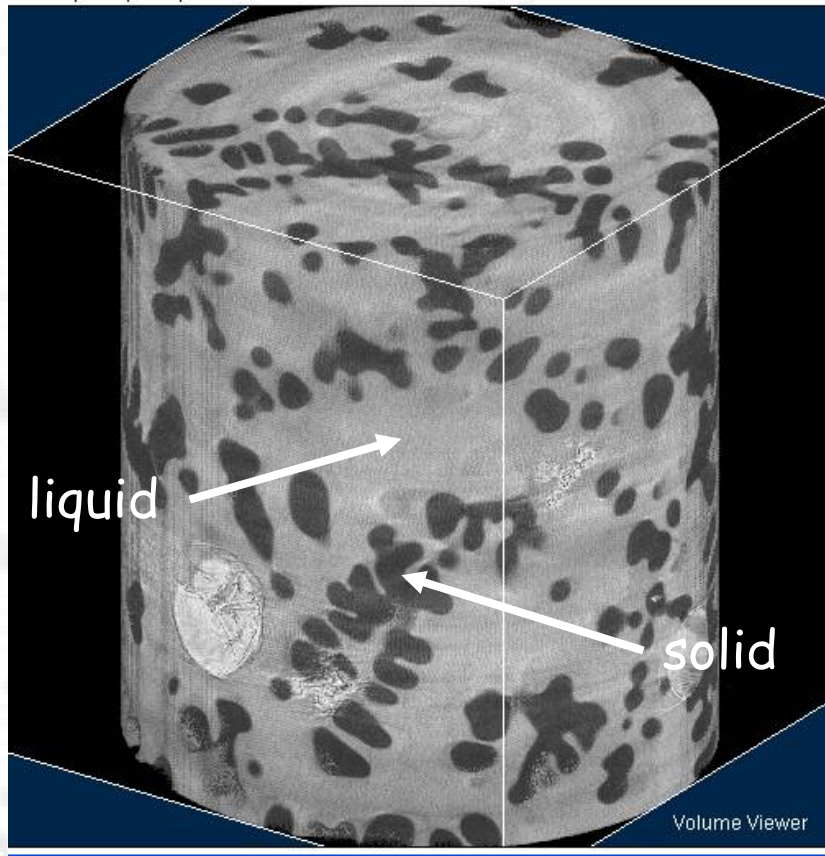


Intermediate case

Conclusion on partial melting

1. Average values of microstructural parameters (particle density, size, S_v) do not follow the standard power laws ($1/3$ or $-1/3$)
2. S_v is proportional to $t^{-1/7}$ (close to Courtney's model for coalescence)
3. Two coarsening mechanisms have been identified: dissolution of small particles to the benefit of bigger ones and coalescence of particles of comparable sizes.
4. Variation with time of average and local microstructural parameters interpreted by considering the two competing coarsening mechanisms

Investigation during solidification



First experiments
on Al-4%Cu

ID 15

Dalsa camera

512x512 pixels

3 μm resolution

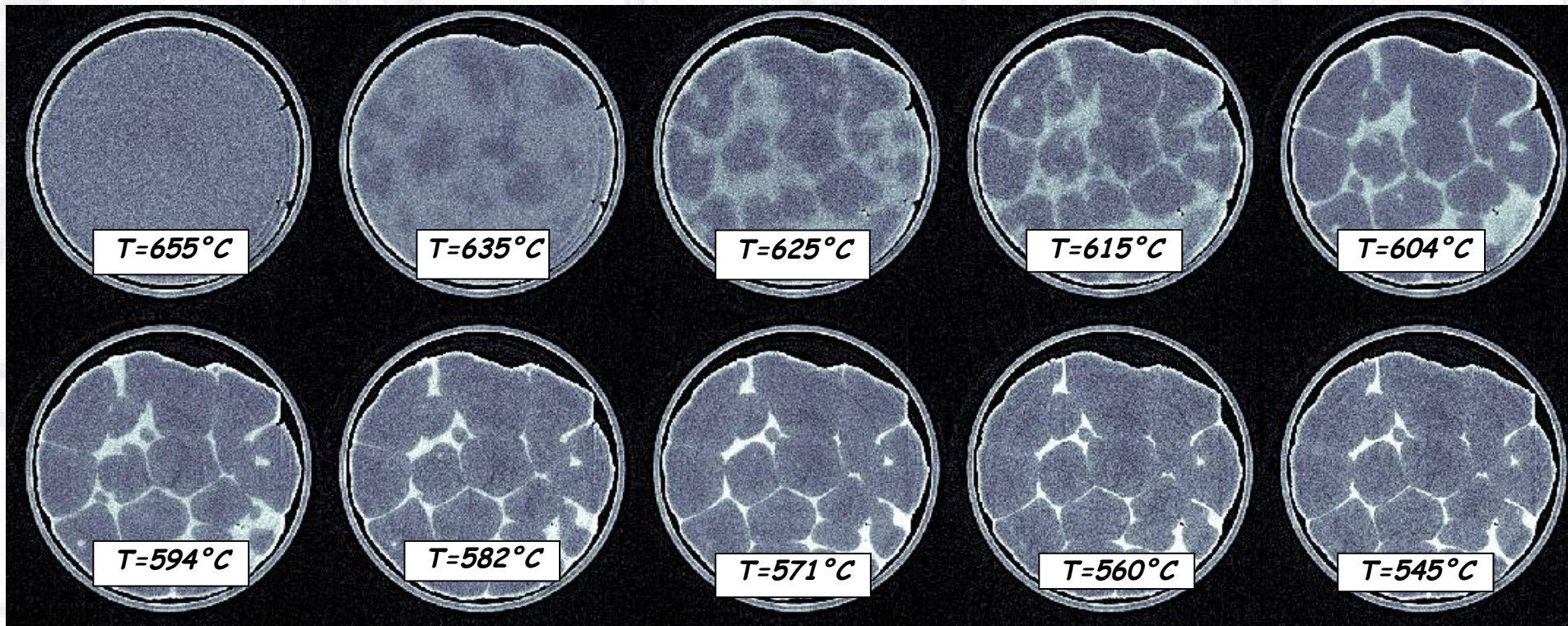
500 projections

Total scan time: 10s

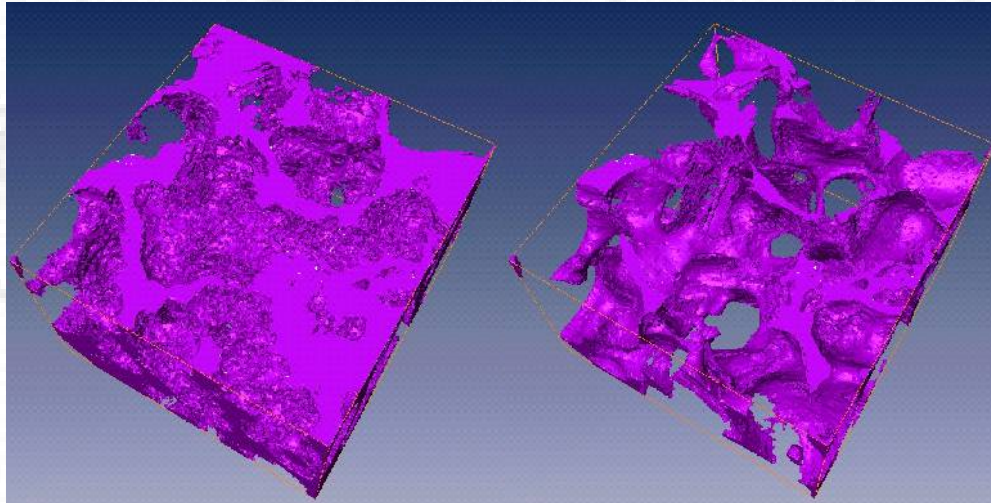
Solidif. rate: 0.1°C/s

Met. Mat. Trans., 36A, 2005, 1515

Evolution of the microstructure during solidification



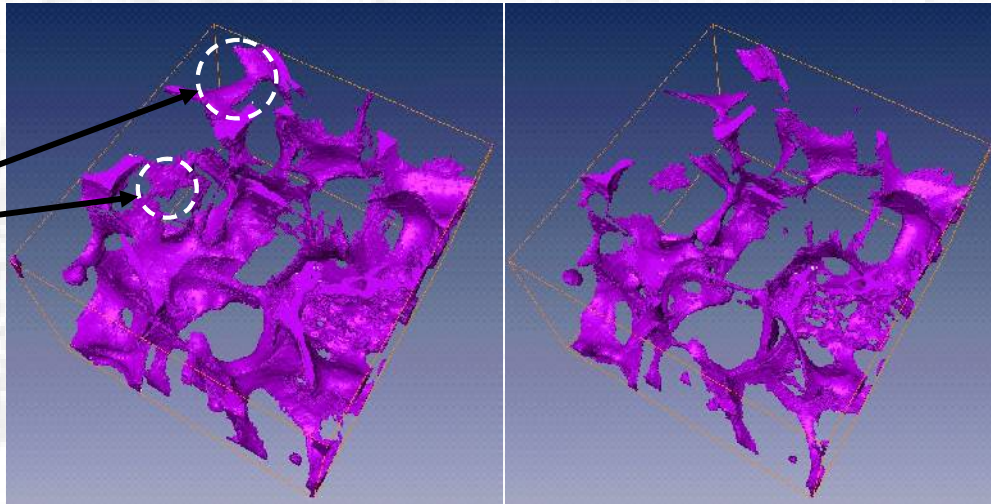
Liquid phase



$T = 625^{\circ}\text{C}$ ($f_S = 0.59$)

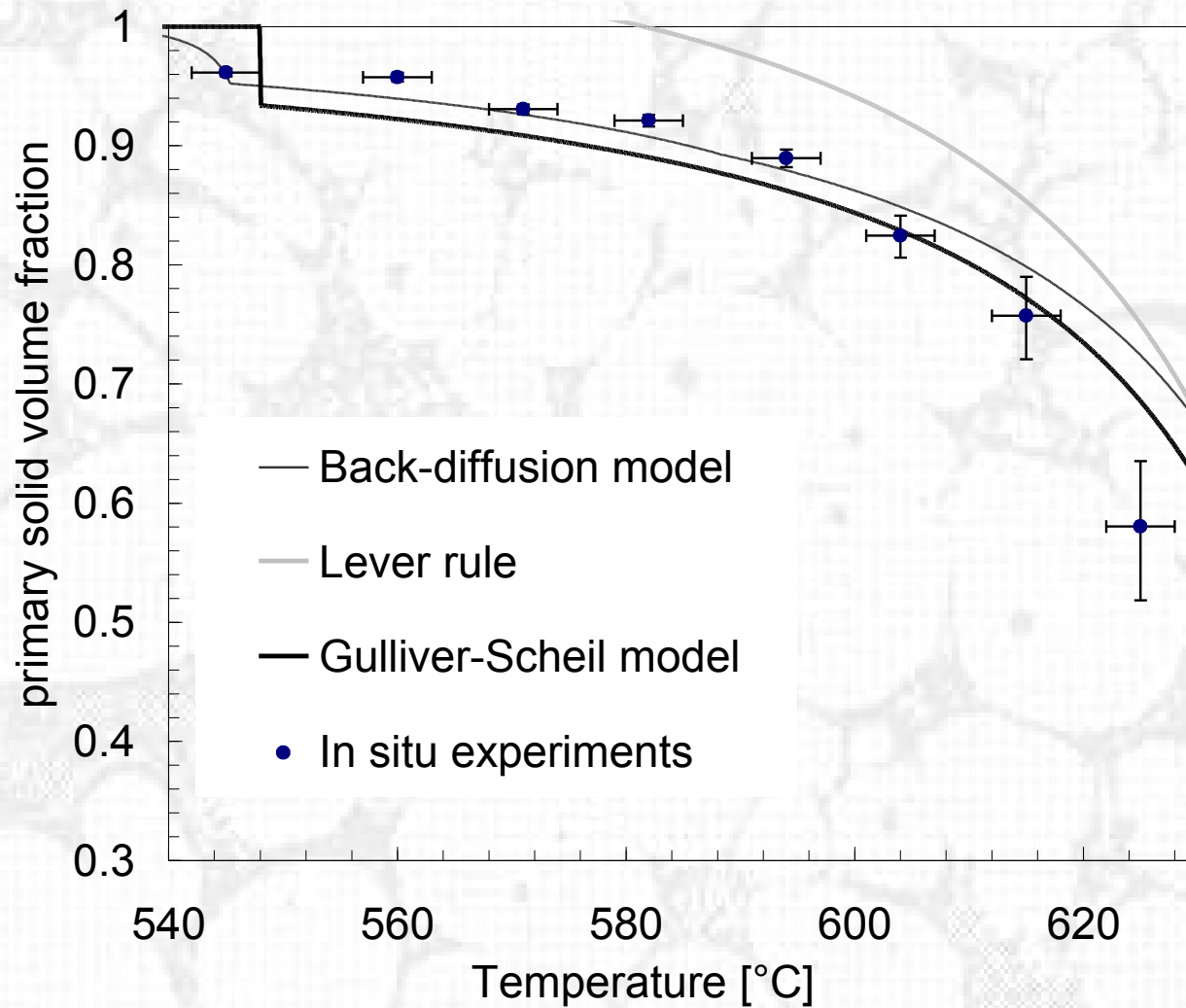
$T = 604^{\circ}\text{C}$ ($f_S = 0.85$)

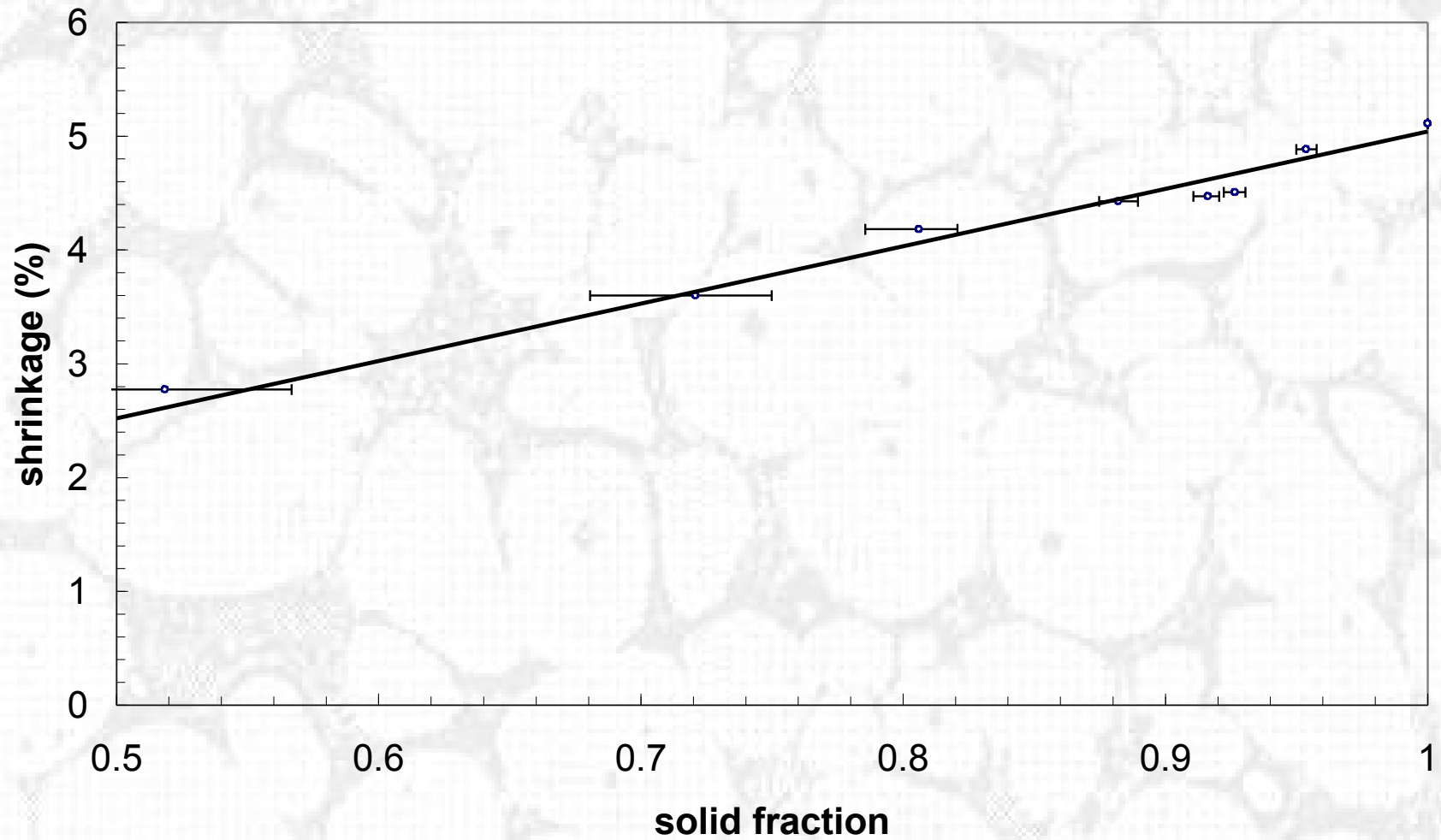
Loss of continuity

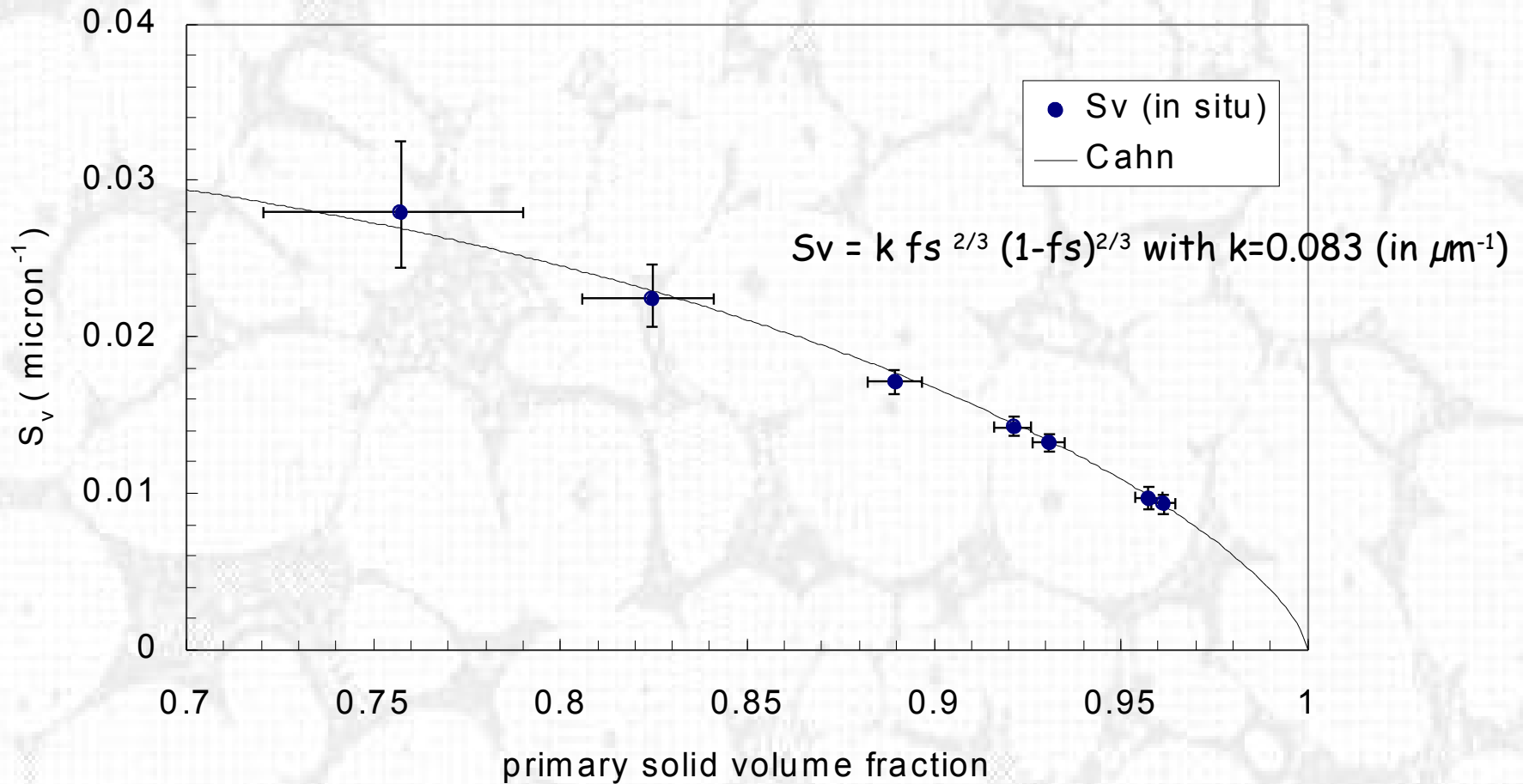


$T = 582^{\circ}\text{C}$ ($f_S = 0.92$)

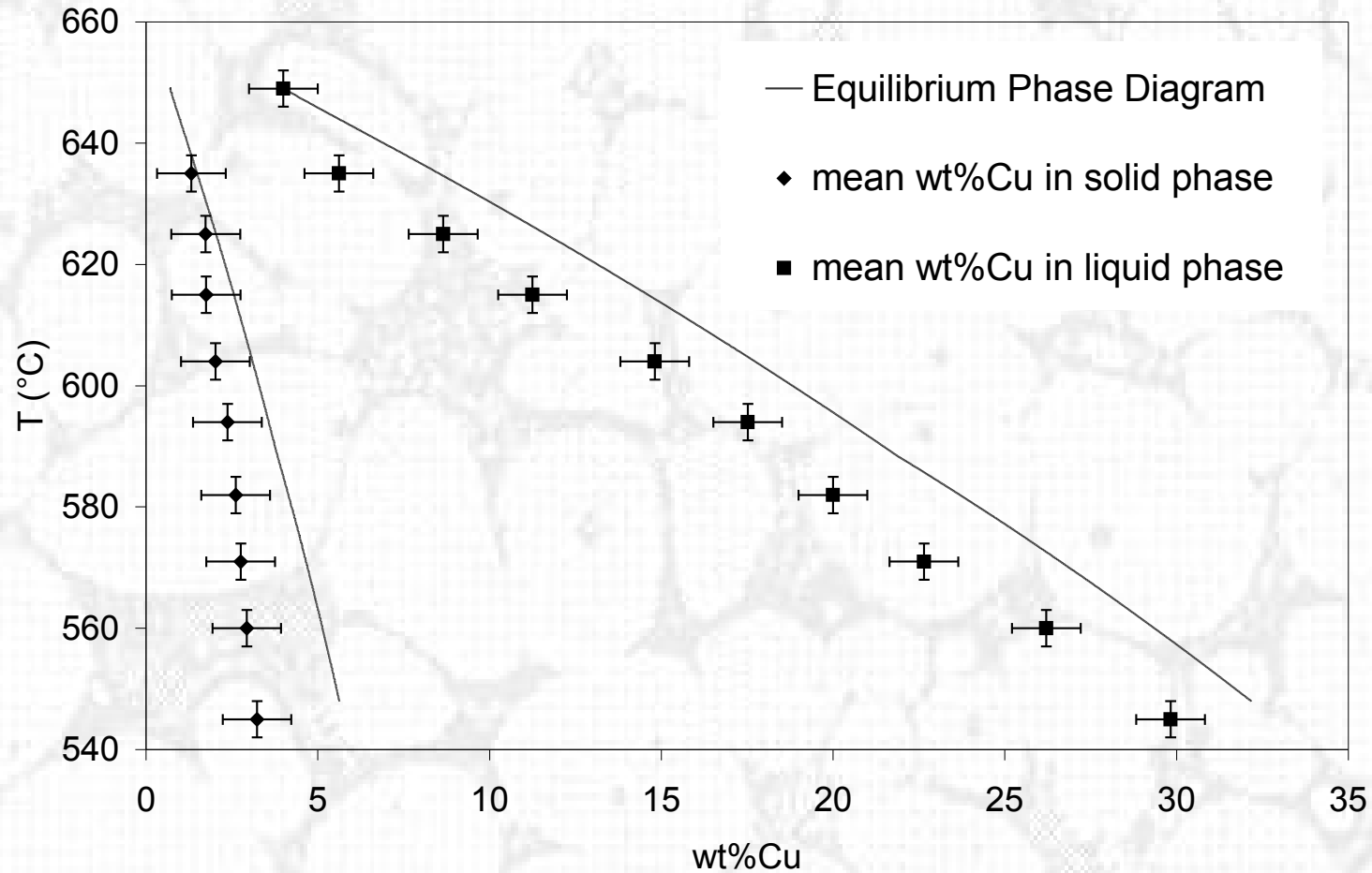
$T = 560^{\circ}\text{C}$ ($f_S = 0.95$)







Calibration of the absorption of the solid and liquid phases



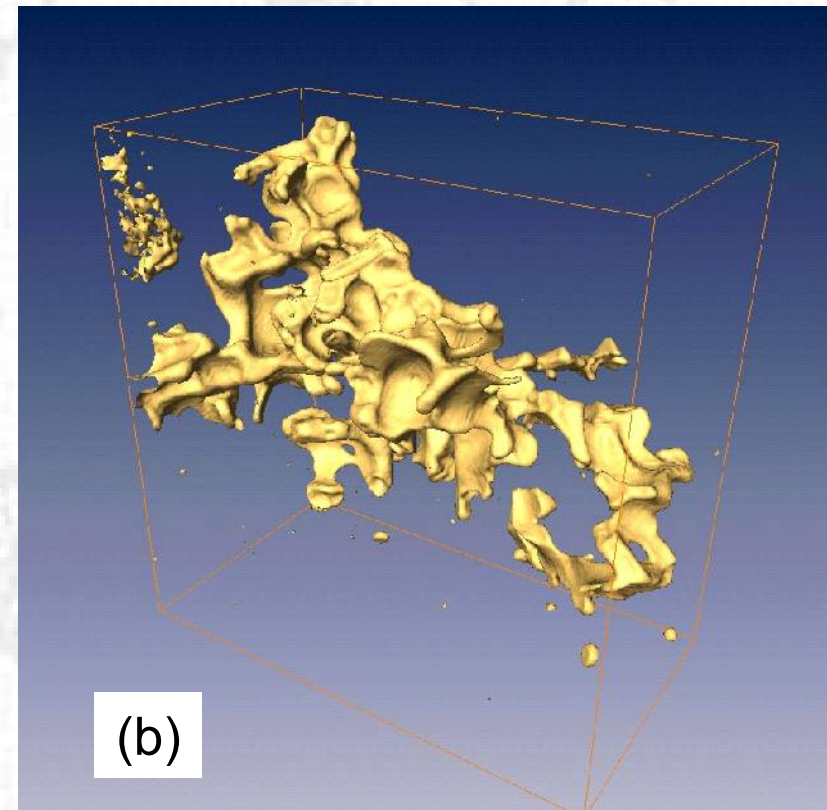
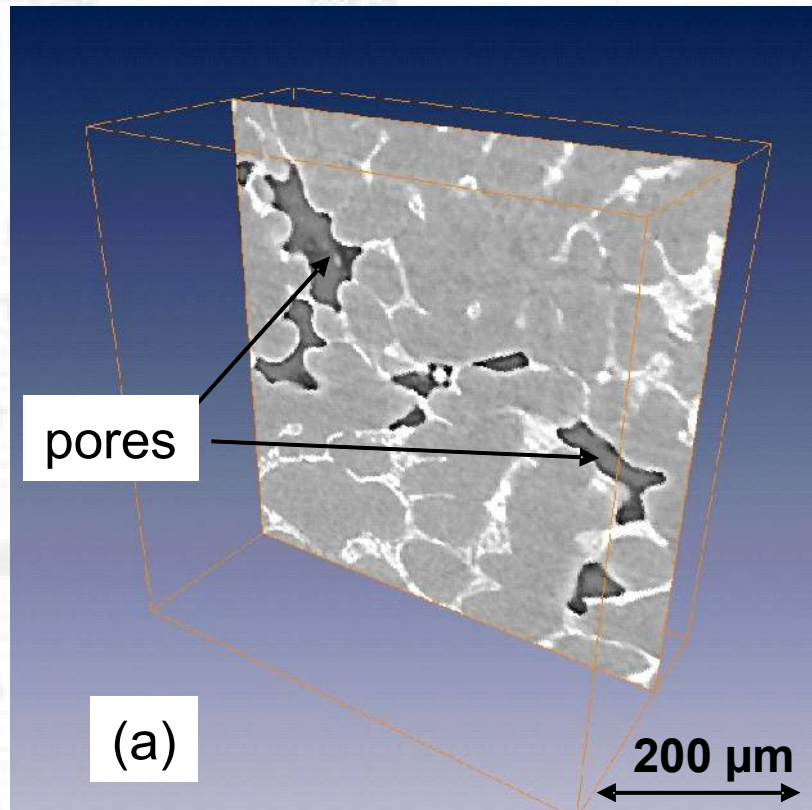
Experiments on Al-10wt%Cu-4.5wt%Si

- development of the dendrites
- formation of the Si phase
- formation of shrinkage pores



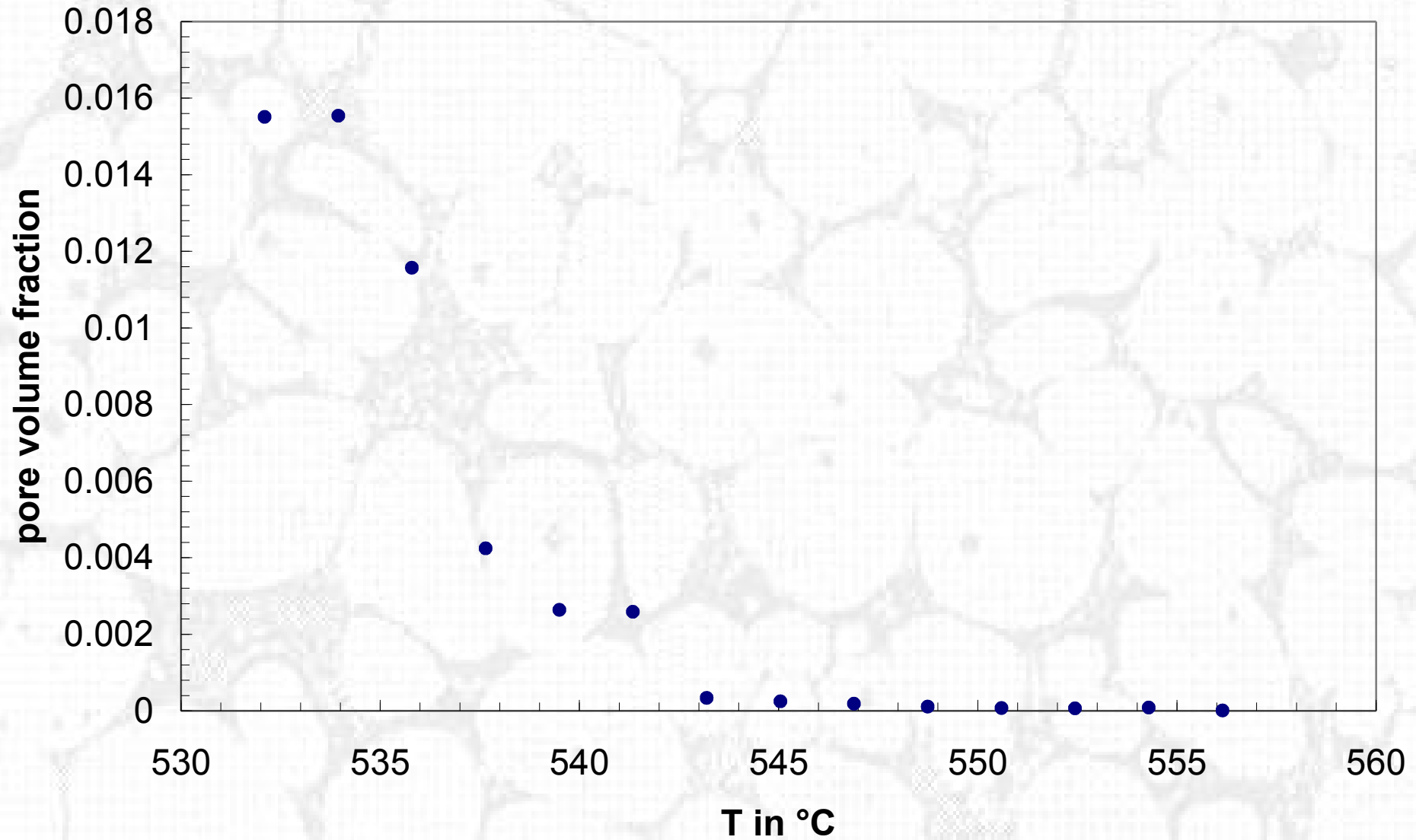
1.5 mm

Observation of pores



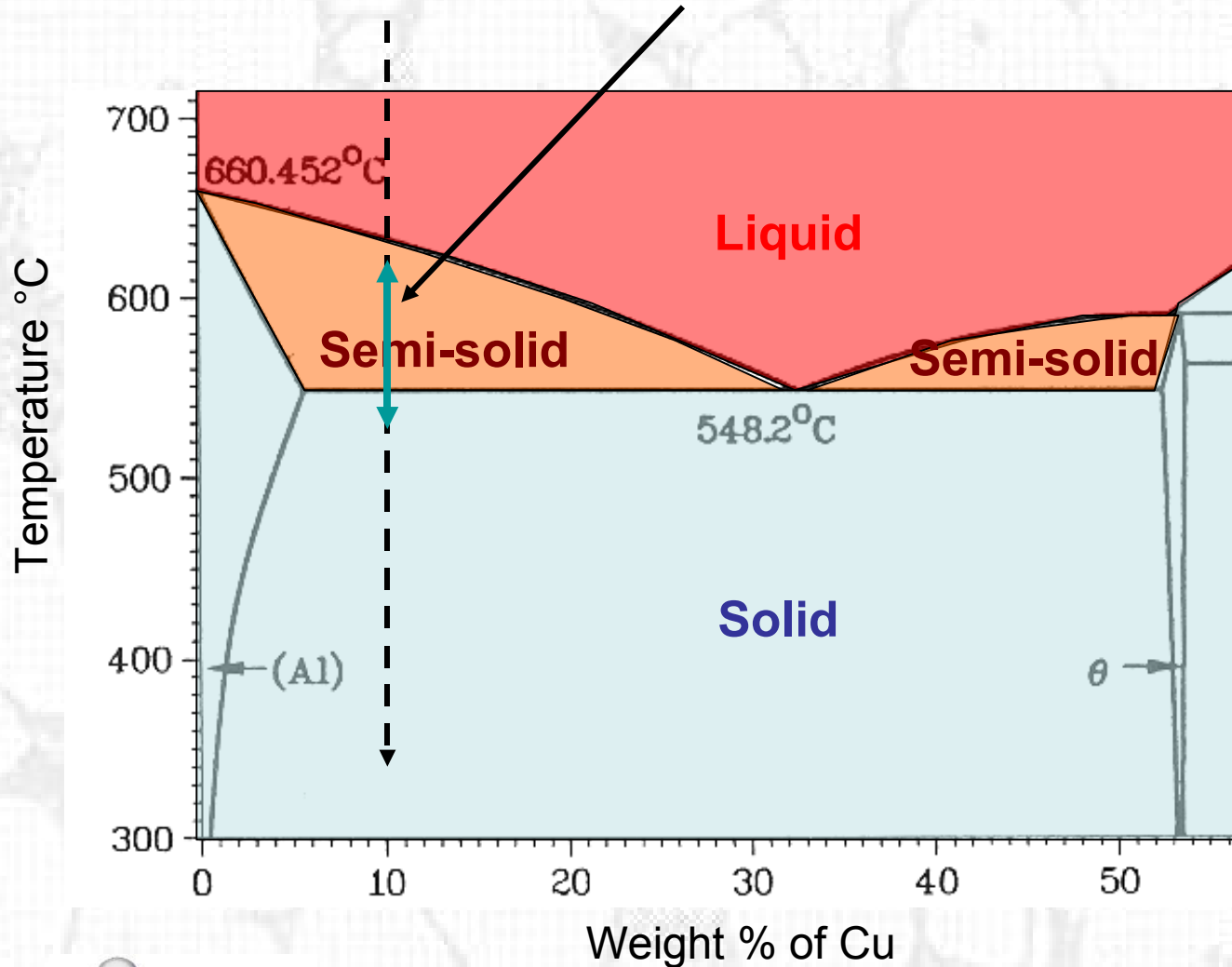
Several pores?

In fact, only **one** complex pore

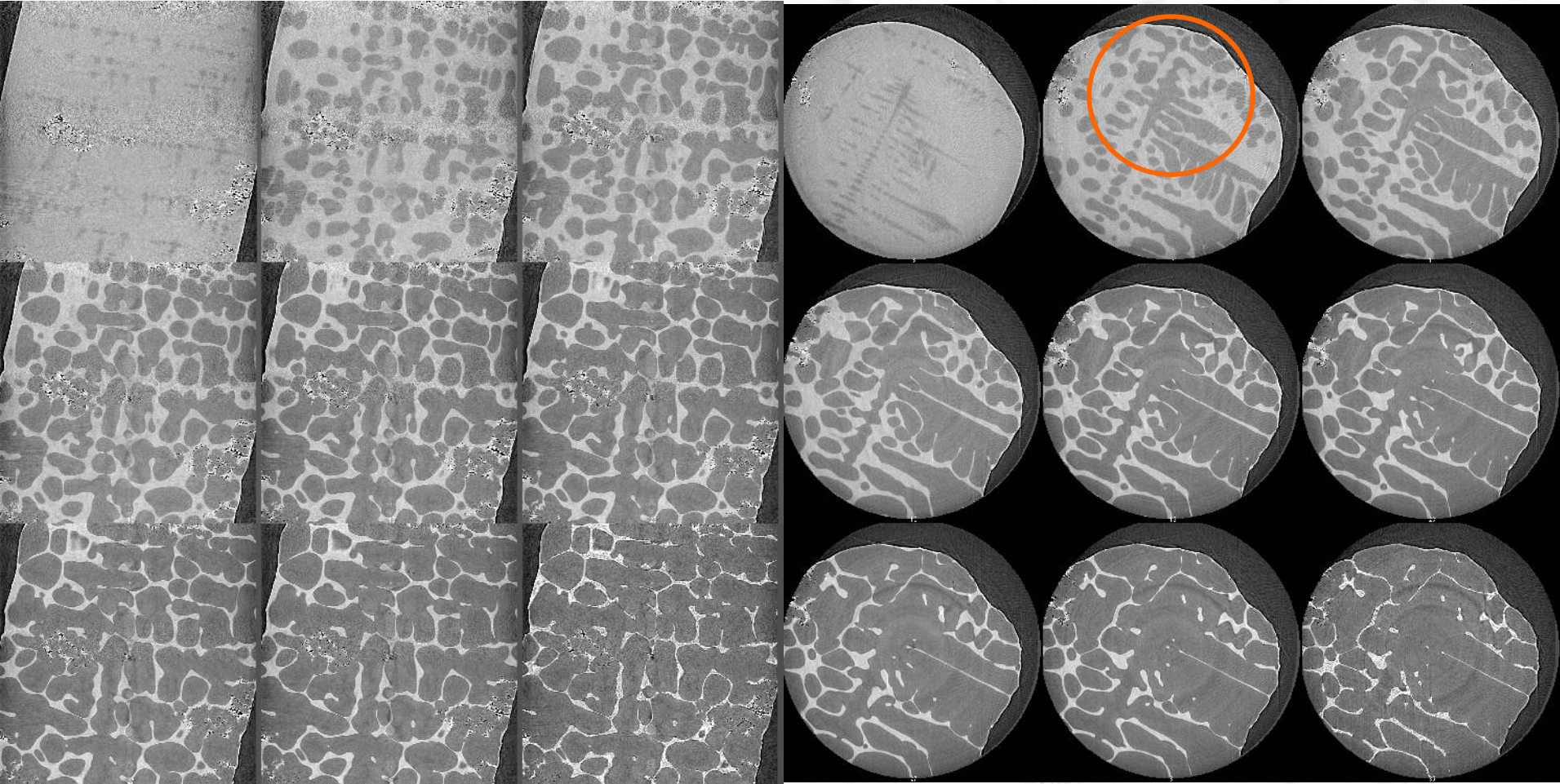


More recent experiments

Al-10%Cu from 628 to 536°C ($T_L=634^\circ\text{C}$)



ID 19
FReLon 2k14
512x512 pixels
Optic 2.8 μm
400 projections
Total scan time 25s
Solidif rate : 0.03°C/C

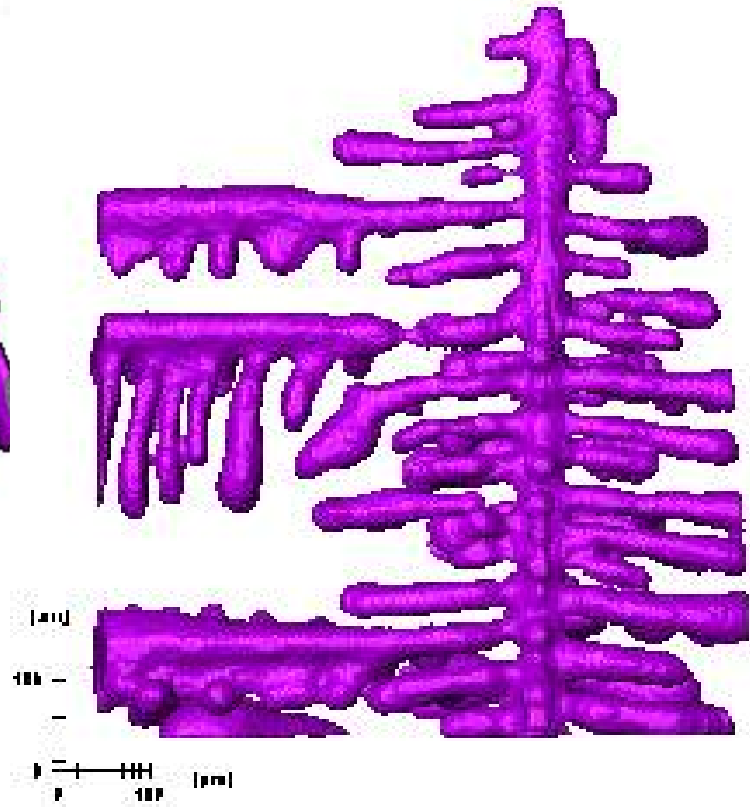
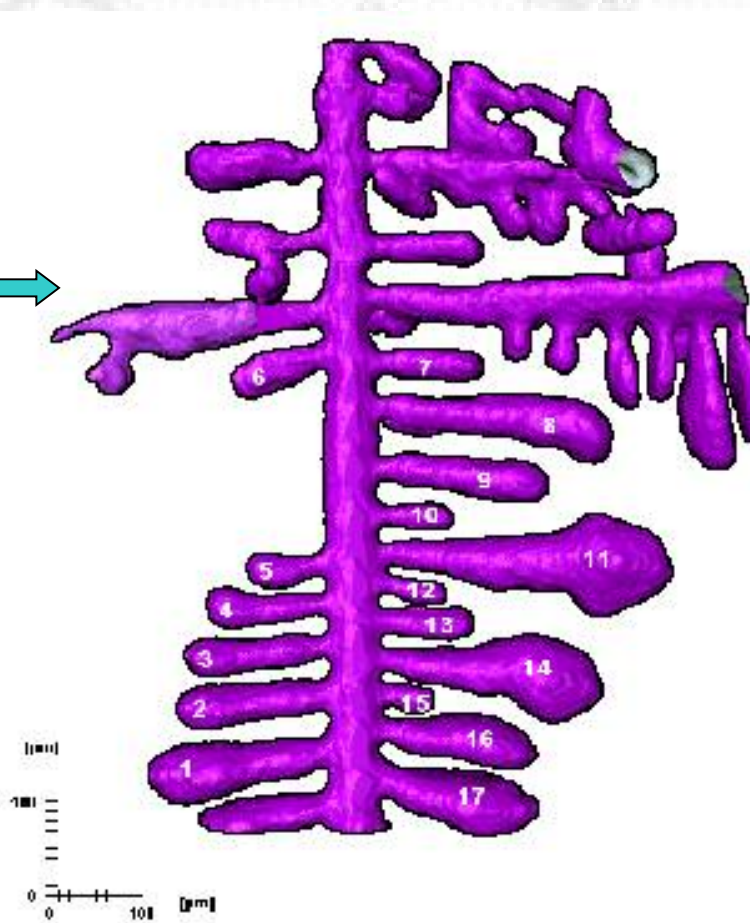


Dendrite 1

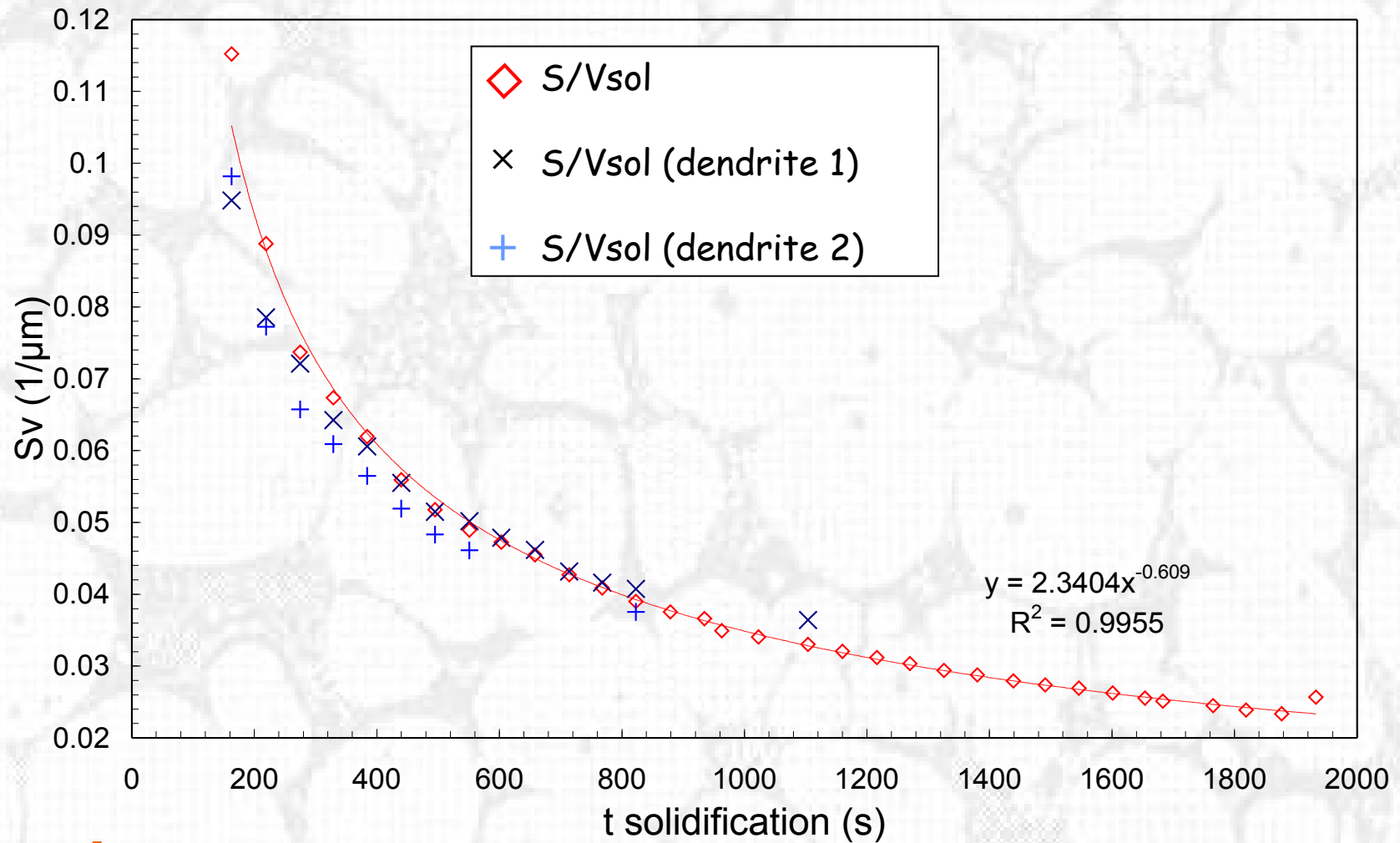
Dendrite 2



(μm)
0 100 200 300 400 500 600 700 800 900 1000



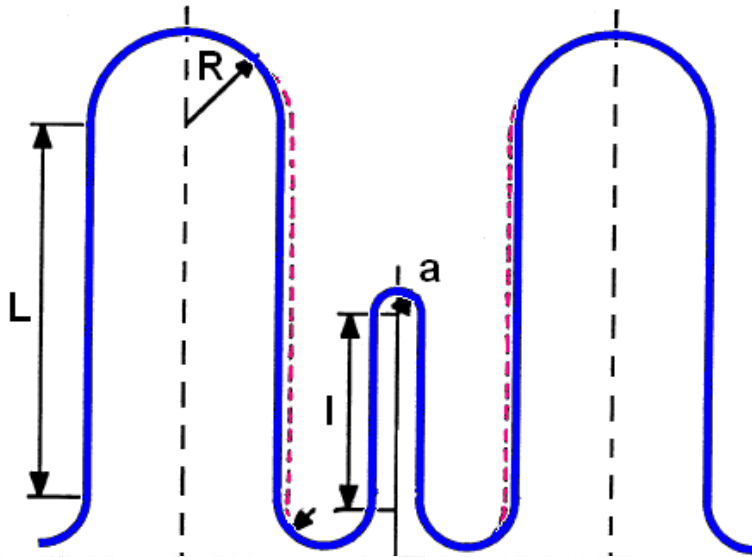
Solid/liquid interface area



For dendrite evolution during solidification, two mechanisms suggested by Kattamis (Chen and Kattamis, *Mat. Sci. Eng.* 1998)

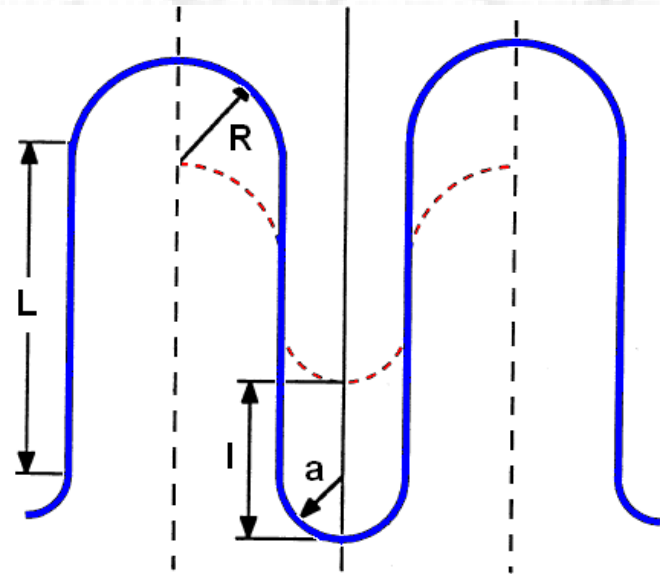
Mechanism A:

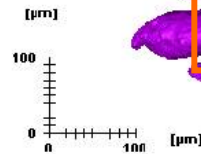
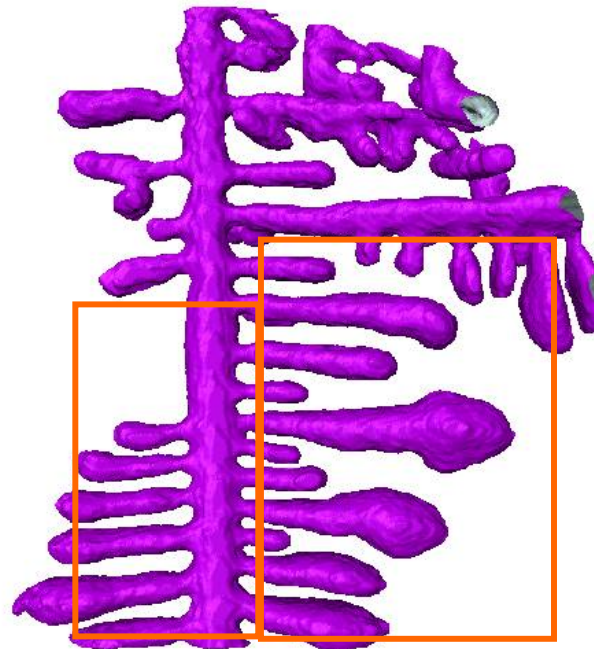
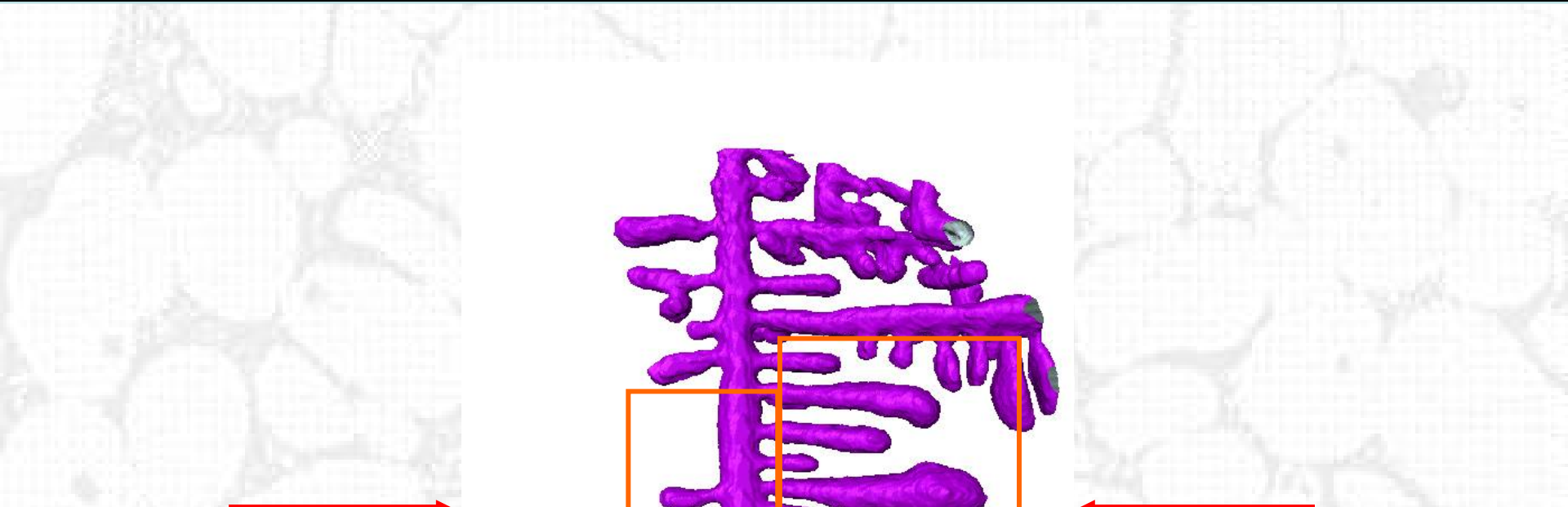
Dissolution of the small dendrite arm



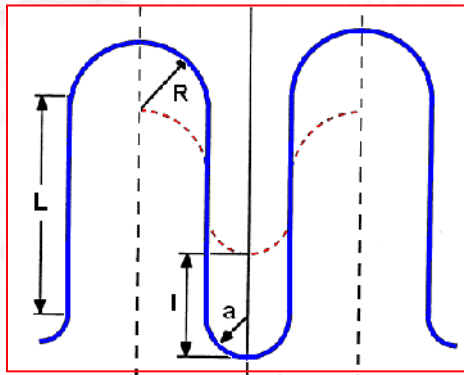
Mechanism B:

Dissolution of the two adjacent arms and deposition in the concave space between the two arms

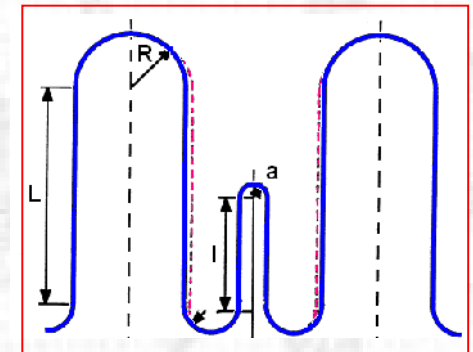




Mechanism B

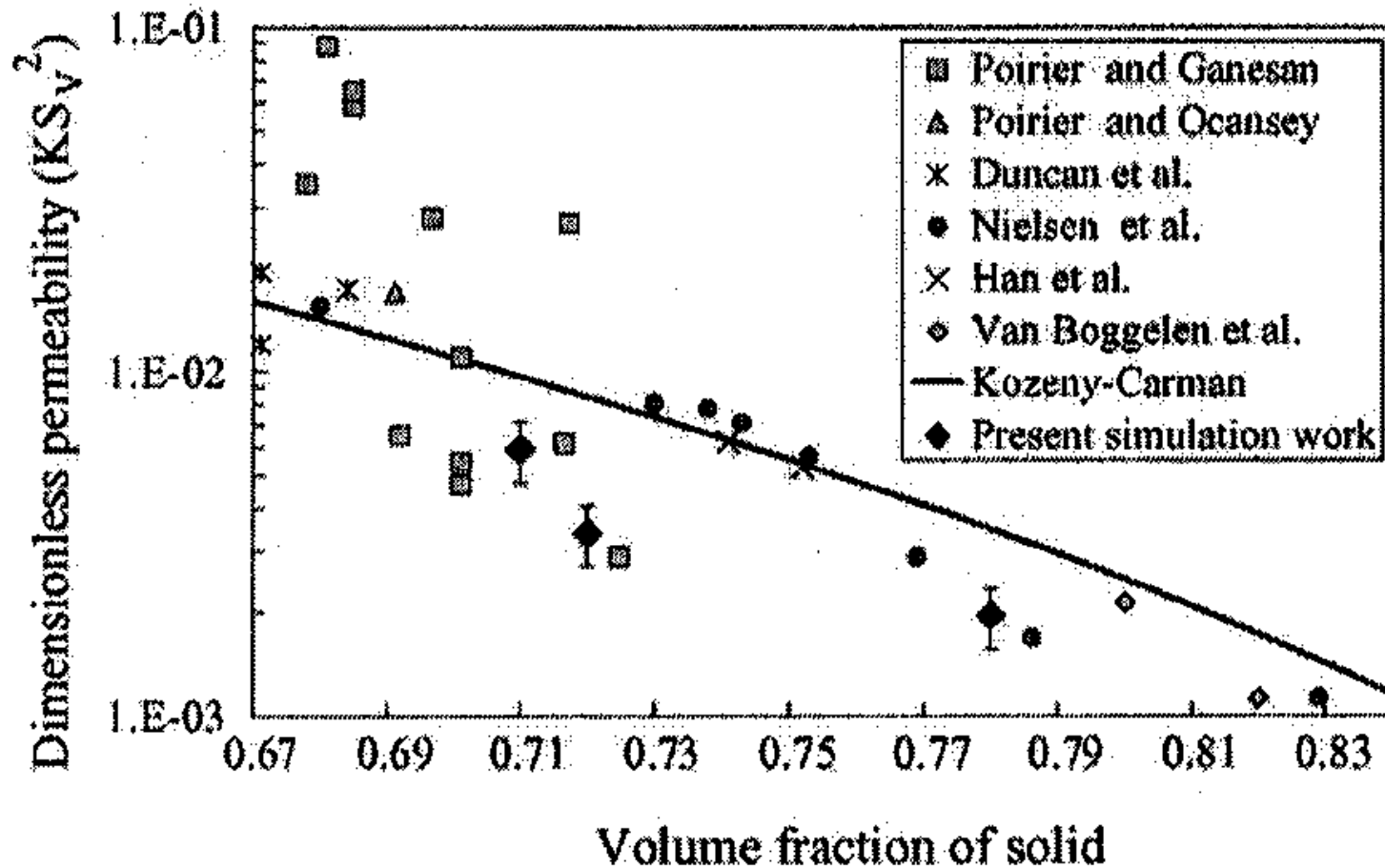


Mechanism A



Conclusion on solidification

1. Microstructural evolution can be quantified during solidification by in-situ X-Ray tomography
2. Parameters such as solid fraction, solid/liquid interface area, mean local curvature of the solid phase, shrinkage, porosity volume fraction can be measured
3. Local observations at the scale of the dendrites allow identifying coarsening mechanisms
4. Tomography volumes can be used to calculate permeability by 3D interdendritic flow simulations



Mat. Sci. Eng. A392, 2005, 112

Conclusion

1. In-situ X-Ray microtomography very powerful to provide 3D data representative of the semi-solid state.
2. Limitations are in terms of:
 - absorption of the solid and liquid phases
 - minimum solid fraction to avoid solid movement
 - cooling rate during solidification
 - specimen size
3. Perspectives
 - deformation during solidification coupled with 3D image correlation
 - improvement in the tomography set-up (resolution, acquisition time...)

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Thank you for your attention

Questions?